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THE MANUFACTURE OF VARNISHES, AND KINDRED INDUSTRIES

LIVACHE AND McINTOSH

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THE MANUFACTURE OF

VARNISHES

AND

KINDRED INDUSTRIES

BASED ON AND INCLUDING THE "DRYING OILS AND VARNISHES"
OF

ACH. LIVACHE

BY

JOHN GEDDES McINTOSH

LATE LECTURER ON VARNISH MANUFACTURE AT THE POLYTECHNIC, REGENT STREET, AND THE BOROUGH POLYTECHNIC

SECOND, GREATLY ENLARGED, ENGLISH EDITION, IN THREE VOLUMES

VOLUME I.

OIL CRUSHING, REFINING AND BOILING
THE MANUFACTURE OF LINOLEUM
PRINTING AND LITHOGRAPHIC INKS
AND
INDIA-RUBBER SUBSTITUTES

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PREFACE TO THE SECOND EDITION

OWING to the extensive enlargements made in this second edition it has been advisable to subdivide the book into three volumes.

The publication of Volumes II. and III. will speedily follow. Each volume, like the present one, will be complete in itself, and no pains have been spared to render them useful, reliable and up-to-date.

No second edition of the work of M. Livache has yet appeared. I am therefore solely responsible for the numerous additions that have been made to both the first and second editions. May the reception of the latter be as cordial as the first.

J. G. M.

LONDON, *June*, 1904.

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UNIV. OF CALIFORNIA

CHAPTER I.

OIL CRUSHING AND REFINING.

Drying Oils.

Definition.—The drying oils are those oils of vegetable origin which, being liquid at the ordinary temperature, possess the property of gradually and progressively absorbing oxygen from the air at the ordinary temperature, and in so doing, instead of yielding a rancid, more or less viscous, greasy mass, change gradually and eventually by insensible gradations from the original condition of a fluid oil into solid elastic substances insoluble in the usual oil solvents.

The greater the quantity of oxygen which such an oil is capable of absorbing in this way, the greater are its drying propensities and properties, and *vice versâ*.

The sequel of changes which such an oil undergoes is termed the *drying* of the oil. As a matter of fact, the oil does not *dry* in the true acceptation of the term. When a substance in ordinary language is said to *dry*, what is meant is that the substance in question loses any adherent, adventitious or interstitial water or hygroscopic moisture, and, in a case of drying of that nature, we can appreciate the change, as it proceeds, both by the eye and the touch. The vapour is visible to the eye as it condenses, and the touch is sensible to the drier feeling as the moisture is expelled to a greater and greater extent. *Such substances, therefore, suffer a diminution in weight corresponding to the amount of water eliminated in the process.* But a phenomenon of quite an opposite nature occurs when a drying oil dries; instead of losing in weight it gradually becomes heavier as the "drying" process proceeds, until the oil has completely "dried," when its weight will be found to have increased some 16 to 17 per cent. In other words, 10 gallons of linseed oil, spread over any given surface, will not only increase the weight of the object on whose surface it was painted by the weight of the 10 gallons of oil, but, when the oil dries, the object will be found to have increased in weight by 110 lb., 93 lb. of which are due to the oil and some 17 lb. due to the absorption of oxygen gas from the air. Now a gallon of oxygen weighs about 95 grains, and as there are 7,000 grains in a pound, and as 17 lb. of oxygen were absorbed, we get a total of 119,000 grains of oxygen absorbed by the oil, which, divided by 95 grains, gives us nearly 1,253 gallons

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of oxygen gas as being required to dry 10 gallons of oil. But the air only contains $\frac{1}{5}$ of its volume of oxygen, so that 6,265 gallons of air are totally deprived of oxygen by the drying of 10 gallons of linseed oil. This is a point in connection with the bad effect of sleeping in newly painted rooms which has not received from the medical faculty the attention it deserves. The headaches and other symptoms may, on investigation, prove not to be due either to the toxic effects of turpentine or to linseed oil emanations, but to the rapid manner in which both diminish the proportion of oxygen in the superincumbent atmosphere by the energetic way in which they absorb oxygen. But there need be no serious effects provided ventilation be attended to. The doing away with fireplaces and heating bed- and sitting-rooms by steam is much to be deprecated. *An ordinary substance in drying loses water and loses in weight. A drying oil in drying absorbs oxygen and increases in weight.* Moreover, if we can see the water being expelled from an object by the clouds of steam which are formed in the process, and although we may increase the rapidity of drying of linseed oil by the application of not too great a heat, such as "drying" the article in a hot-water oven, or a current of air, yet in no instance can vapour be seen to rise from pure and good linseed oil, unless the heat be raised so as to start the destructive distillation of the oil. If the oil contain spirits of turpentine, naphtha, etc., it will of course give off a considerable vapour at a much lower temperature than linseed oil pure and simple.

When linseed oil in its pristine liquid condition is spread over the surface of an object which has been made impervious by the process known as priming, that is to say, its pores have been filled up in a suitable manner, it forms when dried an impervious coating, which still further protects the object from the effects of wear and tear and all those extraneous erosive agencies which we know under the term weather, such as the difference in temperature between day and night, summer and winter, heat and cold, rain, frost and snow.

The Function of Drying Oils as Vehicles for Pigments.—To increase the thickness of the coating, and also to form a harder, more permanent, more anti-corrosive and more anti-erosive layer, the drying oil is generally mixed with substances known as pigments. The oil in drying cements these pigments to the object to which the mixture of oil and pigment is applied. The mixture of oil and pigment is called a paint. The drying oil is said to act as the vehicle. We thus often hear of pigments and vehicles. But a vehicle would be of no use unless it acted as a lasting binding agent as well. Colza oil would form as good a vehicle for pigments as linseed oil, if not better; but as colza oil is a non-drying oil, the paint made by using it would not be a paint at all, as the vehicle would not dry, and thus it would not solidify itself, and hence it could not consolidate the particles of pigment and itself into a hard impervious layer, and the mixture would be more suitable for axle grease than for paint pur-

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poses. In the same way size, the vehicle used in distemper painting, is a good enough vehicle for indoor work when applied to the walls of rooms, but the denuding and erosive action of the weather would render it worse than useless when applied out of doors.

The word vehicle as used by some writers is, therefore, an incomplete definition of the functions of a drying oil when applied in conjunction or in combination with a pigment to the surface of an object. It is the cementing or binding agent, or vehicle, if you will —just as slaked lime is the agent which binds the sand in the mortar to the wall. Not only so, just as the lime cements the particles of sand to themselves and to itself and the agglomerate thus formed to the wall, so also does the linseed oil cement the particles of pigment to each other and to itself and the agglomerate thus formed to the object to which it is applied. But the analogy does not rest here; the slaked lime also increases in weight in drying. Although it gives off moisture it nevertheless at the same time absorbs carbonic acid. For every 18 parts of chemically combined water which mortar gives off in drying and consolidating it absorbs 44 parts of carbonic acid, and consequently increases in weight, as we have just said.

Summary.—The function of a drying oil when used as a vehicle for paints is to so "dry" as to bind and consolidate the pigment in such a way that the combined layer formed by the dried vehicle and pigment, that is, by the dried paint, will withstand the heat of the sun's rays, the erosive action of the weather, and in conjunction with the pigment protect the object to which it is applied from the corrosive and erosive action of the same.

The Function of Drying Oils as Varnish Ingredients, i.e., as "Vehicles" for Resins.—The chief function of drying oils as ingredients of varnishes is first of all to dissolve the resin so as to bring it into the fluid condition; here again colza and other oils would act similarly, but the solution of resin in colza oil would never dry, and the only use for such a product would be for axle grease, and even for that purpose it would not be very fit, and not only so, it would be a very costly way of making axle grease. Here again the function of the linseed oil is to dry, and in so doing to cement together the particles of resin by an elastic binding agent consisting of the product into which the linseed oil is resolved on drying. The greater the quantity of linseed oil and the fewer the particles of resin which it has to cement together, and which are present in a "dried" coating of varnish, the more elastic and durable will be that coating. If a linseed oil substitute consisting of hydrocarbides, such as rosin oil, be substituted for a drying oil, *e.g.*, linseed oil, whether in a paint or varnish, this durability and elasticity is lost, the dried product dissolves in weak solutions of alkali, and even in warm solutions of household soap, as is seen in the case of the front door of the housewife who must always be scrubbing. Her newly painted and varnished front door in a few months looks as if it had not been

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painted or varnished for as many years. Moreover, the melting-point of the dried product of linseed oil is very high, as any one may satisfy himself by trying to melt linseed oil skins, but the melting-point of the "dried" (*sic*) product of hydrocarbide oils, *e.g.*, rosin oil, is no better than that of common rosin. Moreover, the dried product of such oils or varnishes is soluble in the original varnish, especially in the sun, when the former melts, a fact which gives rise to many exasperating difficulties when it is desired to apply a second coating of the same paint or varnish on the same article. In the cool of the evening the coating may be as "dry as a bone," in the heat of the day it is simply a liquid pitch, and if the coating does eventually so dry and harden as to withstand the heat of the sun, it is such a mass of cracks and furrows that those on an elephant's hide might well be compared to them. These cracks and furrows are produced by the difference in temperature between day and night, producing hundreds of alternate liquefactions and solidifications of the coating, into the composition of which this linseed oil substitute enters so largely. Moreover, in the cool of the evening the coating seems to dry, but it is merely surface drying, underneath is a layer of liquid pitch like that of a lake of asphaltum, and it only requires the heat of the sun to melt the surface crust to liberate streams of the liquid confined beneath.

The great function and characteristic property of a drying oil is to yield on drying a coating of high melting-point, great elasticity, great imperviousness and great durability, and resisting both natural and artificial reagents and solvents.

Linseed oil substitutes have one advantage: they are easily removed when necessary, and, in their case, one need not go far to find a paint remover. There is no necessity to burn it off, as in the case of good genuine linseed oil. Were the latter more used there would not be such a market for the numerous brands of more or less efficacious "paint removers".

The drying oils employed by varnish manufacturers are (1) linseed oil, from *Linum usitatissimum* (the flax plant); (2) poppy-seed oil, from the *Papaver somniferum* (the opium poppy); (3) walnut oil, from *Juglans regia;* (4) hemp-seed oil, from *Cannabis sativa;* castor oil, from *Ricinus communis*, is sometimes used for special purposes. Amongst other oils occasionally used, suggested to be used, or said to be actually used, are maize oil, sunflower-seed oil, safflower-seed oil and some others. Indeed many other kinds of drying oils occur in the different organs of various plants, but they are but little used in actual practice; amongst these are cucumber oil, from *Cucumus sativus;* grape-seed oil, from *Vitis vinifera;* bankoul oil, from *Aleurites triloba.* It is necessary, however, to make special mention of the quick-drying oil *Paulownie of Japan*, the fine oil of the *Dipterocarpus crispaletus*, the *Kai-dan-long* of the Annamites, and *Elæococca*, Japanese wood oil, the best drying oil known.

The study of drying oils will, in this small treatise, be confined principally to *linseed oil*, the drying oil *par excellence.*

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Linseed Oil.

The flax plant *Linum usitatissimum* is a native of Europe and the north of Africa, and is said to be indigenous to Britain. The flax plant has been cultivated from very ancient times. We read of its having been grown by the Israelites so far back as the time they were in Egypt. Flax is cultivated for a double object, firstly, for the sake of its stem, which yields by maceration the tenacious fibres used in the manufacture of linen; and, secondly, for the sake of its seeds, which by expression, or extraction by means of solvents, yield linseed oil.

Flax loves a deep, friable, rather moist soil. It grows well upon land newly broken up from pasture. In Ireland it is usually sown by the small farmers after potatoes. A fine, nicely pulverised seed-bed is essential, and this is rolled after the seed is sown. The usual time of sowing is from the middle of March to the end of April. Dutch seed is in the highest estimation, affording a greater produce than the American seed, and a finer quality than the Riga. The after cultivation consists in taking up the weeds by the hand—the only way in which it can be done, and a tedious process if the land be at all foul. The crop is raised by pulling up the plant by the roots. The produce in seed is from 6 to 8, and sometimes as high as 10 or 12, bushels per acre. The best of it is kept for sowing again, and the next quality is crushed for oil. Flax, when allowed to ripen its seeds, is a very exhaustive crop, even more so than corn, and in most parts of Britain is so uncertain that its cultivation has hitherto been very limited. But a great deal of flax was at one time raised in Ireland Its cultural zone is almost world-wide.

The linseed imported into England comes chiefly from India and Russia, the former being known as East Indian and the Russian as Baltic seed. The colder the climate in which the seed is grown the greater are the drying properties of the oil, but the colour is worse. In India white seed is preferred not only because it yields more oil, about 2 per cent., but also because it yields it more freely than red seed, and further it furnishes a softer and sweeter cake, which is better relished by cattle than the cake from white seed.

White seed comes to market largely adulterated with hemp and rape seed. The latter being difficult of separation, greatly depreciates the market value of the linseed. La Plata seed is impure and oil bad.

"Next to cotton-seed oil," says a reliable American authority, "the most important of the United States of America oils is that made from linseed. It is only within the past twenty years that the cultivation of flax in this country has been of sufficient consequence to make the domestic crop of seed come anywhere near supplying the demands of the crushers. Flax has been grown in many of the middle and Western States for years, but it was not until the broad acres of the North-West and the States beyond the Missouri were turned to its cultivation that the domestic crop assumed such proportions as

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to gradually crowd out the imported. Until about 1880 the American crushers had to depend upon the Calcutta or Argentine seed, although the small mills located here and there through the country indicated where local supplies of seed were to be had from domestic growers of flax. As the domestic flax is not used for making linen, but is grown only for the seed, it is evident that the farmers would not have turned their attention to it but for the bounty which was offered in a duty of 25 cents per bushel, and even this heavy tax did not stimulate them to great activity in this direction until prices on wheat and corn began to feel the effects of excessive production. At the present time the duty of 25 cents per bushel is prohibitory except when seed is being manipulated by speculators. Instead of being an importing nation the United States of America have come to be exporters under ordinary conditions. The flax-seed crop this year is estimated at 19,000,000 bushels, a large portion of which will be exported, provided prices are not kept at the high prices now existing. The exports of the seed for last season were 2,500,000 bushels. There has been a large fluctuation in prices. In my earlier days, as a broker, I sold many thousand gallons at 1 dollar 50 cents and 1 dollar 75 cents per gallon of 8 lb., and of late years it has sold down around 27 cents up to 51 cents at the present time. Linseed oil is used chiefly in paints, but in Great Britain it finds its way into the soap kettles at times when the price is low, although it is not ranked equal to some other oils and fats by soapmakers. As yet the price has never gone low enough here to make linseed oil a competitor of the better-known soap greases, although in 1897 it could have been put into the Western soap kettle at a trifle over 3 cents per lb."

Linseed should be kept three to four months, in a dry place, before expressing the oil, as the oil furnished afterwards is much more abundant than when expressed immediately after harvest. Oil from seed which has not been matured in this way is watery, and the more so if the seed were not ripe at the time of harvesting.

The seeds of the flax plant (linseed) consist of small, bright, greyish-brown, slippery, elongated bodies, containing a mealy oleaginous albumen.

The chemical composition of the seed is on an average as follows:—

COMPOSITION OF LINSEED.

	Per cent.
Water	7·50
Oil	34·00
Albuminoids	24·44
Carbohydrates (starch, mucilage, fibre, etc.)	30·73
Ash	3·33
	100·00

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The composition of the different parts of the seed is as under:—

Episperm	21	Gum and soluble salts	14
		Soft resin and fixed oil	1
		Water	2
		Matter insoluble in water and ether	4
Endosperm	23	Soft resin and fixed oil	6
		Water	2
		Matter soluble in water	3
		„ insoluble in water and ether	12
Kernel	56	Fixed oil	30
		Water	5
		Matter soluble in water	3
		„ insoluble in water and ether	18
	100		100

The husk of linseed is of a purplish-brown colour, and, as will be seen from the above analysis, contains much less oil than the kernel or nucleus. The kernel is yellowish white in colour, and the richest in oil. By expression in the cold linseed yields from 21 to 22 per cent. of its weight of oil. By the combined action of heat and more powerful pressure as much as 28 per cent. may be obtained. But if a pure oil is desired expression in the cold is much to be preferred, as there is then less tendency to dissolve the mucilage. Expression by steam, however, yields a very good oil when the steam heat does not exceed 200° F. The usual way of getting rid of the large amount of mucilage is by steam heating, after which the seeds are bruised and pressed. The marc remaining after expression is generally known as oil-cake, and is an article of great value to the agriculturist, not only as a direct article of food for cattle, etc., but as an indirect way of enriching the soil. A farmer by the consumption of a ton of linseed cake on his farm enriches the soil to the same extent as if he had spent a very considerable sum on chemical manures.

Linseed oil-cake has the following average composition:—

	Per cent.
Water	7 to 10
Albuminoids	22 „ 30
Oil	9 „ 13
Carbohydrates	30 „ 36
Fibre	8 „ 10
Ash	5 „ 8

The higher the temperature and the more powerful the pressure used to express the oil from the seed the less valuable is the cake. The oil varies in genuine cakes from 8·5 to 13 per cent., but is generally from 10 to 11·5 per cent. The albuminoids vary from 25 to 32 per cent., but on an average are about 26 per cent. The ash ranges from 5 to 9 per cent., but is usually from 5·8 to 7 per cent. Good linseed cake

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has a reddish-brown colour, uniform appearance and pleasant mild flavour and odour. Very dark cakes are generally over-heated, and very white ones adulterated. Oil-cake is said not to be a good food for pigs as it renders their flesh soft and oily.

The Crushing of Linseed.

Rolls.—The first process in crushing linseed is to pass it through the rolls (Fig. 1). It is conveyed to the hopper by an elevator or chute, and is distributed to the rolls (Fig. 1) by a fluted feed roll the same length as the feed rolls at the bottom of the hopper. From the feed roll it falls on a guide-plate, which carries it between the *first* and *second* rolls. After passing between these rolls and being partially crushed it falls on a guide-plate on the other side, which carries it between the *second* and *third* rolls, where it is crushed more fully. It then falls on another guide-plate, which carries it between the *third* and *fourth* rolls, where it is ground more fully still. In the larger rolls it falls on a fourth guide-plate, where it is conveyed between the *fourth* and *fifth* rolls and receives the final grinding. The seed is thus crushed four times in its passage through the rolls, which, being brought into contact by a combination of a screw and spring, give a smooth and easily regulated pressure. When the seed falls from the bottom roll the grinding is found to be much more complete and perfect than in seed that has passed through rolls and under stones of the old description.

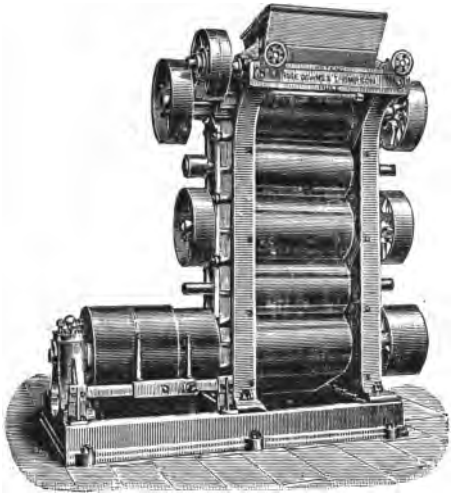

Fig. 1.—Rolls for Crushing Linseed.

Steam-Heating Kettle and Moulding Machine.—After passing through the rolls the seed falls into a screw conveyer, which places it in an elevator that raises it to the steam-jacketed kettle (Fig. 2, A), where the seed is heated and damped by a jet of steam and agitated by a revolving stirrer. These kettles are made of cast-iron, and are constructed in the strongest and best manner. There is only one steam joint in them, and that is faced in a lathe or planed quite true. There is, therefore, little liability to leakage—always a dreadful nuisance. When lagged, the kettle body is fitted with a wooden frame, covered with felt, and the felt is enclosed in iron sheeting. Up to this point the operations have been automatic. With the

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kettle is worked the moulding machine (Fig. 2, B). A man measures out sufficient seed for a cake, forms it of the proper shape, cases it in a strip of bagging, and passes it to the presses.

Hydraulic Presses.—Each press holds 16 cakes, and is furnished with steel cylinders 16 inches in diameter, which carry a working pressure of 1¾ tons on each square inch of the ram. All the columns, cylinders, rams and heads are planed and turned accurately to gauges, so as to ensure that every part will take its due proportion of strain and no more; the pockets that take the columns are not cast, as is usual, with fitting strips top and bottom, but are all solid throughout, and are all machined out of the solid to gauges. This method of treating the pockets has the effect of almost entirely preventing the columns from breaking. In certain types of presses the pockets for the columns are bored and the columns are fitted with steel nuts. The plates between which the seed is pressed are all well fitted and have a corrugated surface. If a brand be required on the cake as, say,

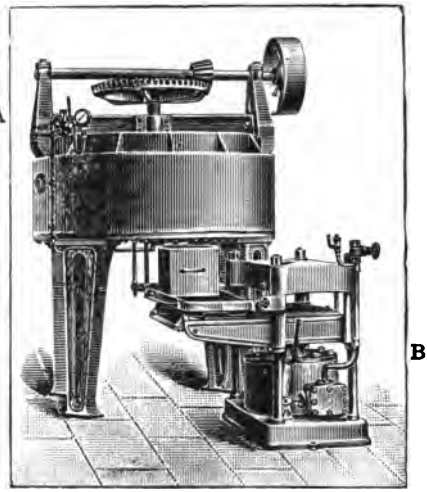

FIG. 2.—A, Steam Kettle; B, Moulding Machine.

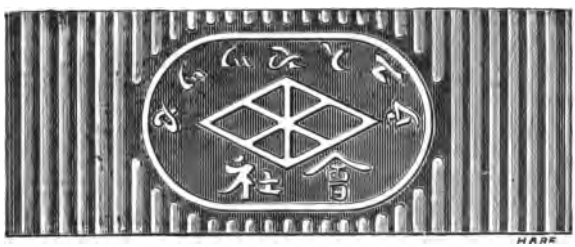

FIG. 3.

letters about 3 inches long are clearly cut into one side of the plate and all the remainder is corrugated. These plates are all made of wrought and cast malleable iron, and the columns are made of best scrap iron or mild steel. The cylinder is crucible cast steel.

Pumps.—The pumps are made of the highest class of crucible cast steel and are all bored out of the solid. Two of the pump rams are 2½ inches in diameter and have a stroke of 7 inches. These rams give only a limited pressure, and the arrangements are such as to

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give the limited pressure on each press in about fourteen seconds. The pumps then stop automatically. The work is then taken up by two other pumps having rams 1 inch in diameter and a stroke of 7 inches, and is continued by them until a gross pressure of 2 tons per square inch is attained. This is the maximum and is arrived at in less than a minute. The oil as it comes from the presses falls into the tank underneath, which serves as a foundation for the presses, and is pumped from there into the store cisterns.

Cake Paring and Moulding Machine.—The cakes require paring. The parer strips his cake and lays a number of these on a stool or table. He takes up one and lays it on the machine (Fig. 5), having one side parallel to the trough and overlapping it about an inch, one end being placed against a fence, either to the right or left, depending on what stroke the machine is taking. The knife block passes along and cuts one side clean and straight. The cake is now turned over and the other edge treated in a similar manner. He now turns the cake half round, places one side against a fence, and cuts off the oily part at one end, then reverses the cake and does the same with the other end. The cake is now ready for the market, and has been pared by two double strokes of the knives, the speed of which is about thirty per minute. Fences or gauges may be added to give all the cakes one exact length and breadth, but these make more parings, as the fences have to be set to suit the worst cakes. These machines can be driven by a shaft either parallel with or at right angles to the knife bar, and are suitable for paring either parallel or taper cakes.

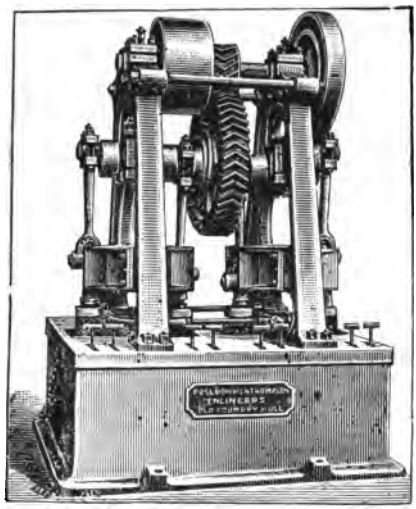

FIG. 4.—Pumps.

Edge Runners for Grinding Cake Parings.—The parings are taken to the edge runners (Fig. 6) to be ground. These are made of the best Derbyshire grit stone, and are furnished with a set of cast-iron centres. One centre has a long boss on it, which passes right through the stone and the centre on the opposite side, and has a long brass fixed on each end of it. These centres are securely fixed to the stones with bolts that pass through them, and they are also run into the stones with lead. On the outside centre there is a cap fixed with screws, quite oil-tight, and large enough to hold several pints of oil. The spindle on which the stone revolves passes through the centre of the upright shaft, and the stone centre has a cap fixed on each end of it with cotters. These caps are enclosed in the oil-cups

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described above, and revolve among the oil contained in them, thus causing such a perfect lubrication of the spindle as to make it quite unnecessary to withdraw it oftener than once in several months. The casting that carries the vertical shaft passes through the bed-stone and goes beneath it, projecting beyond its circumference at six points, where it is attached to the circular pan above by six brackets. The top of this casting is fitted with a "step," constructed in several parts, which can be removed or replaced in a few minutes

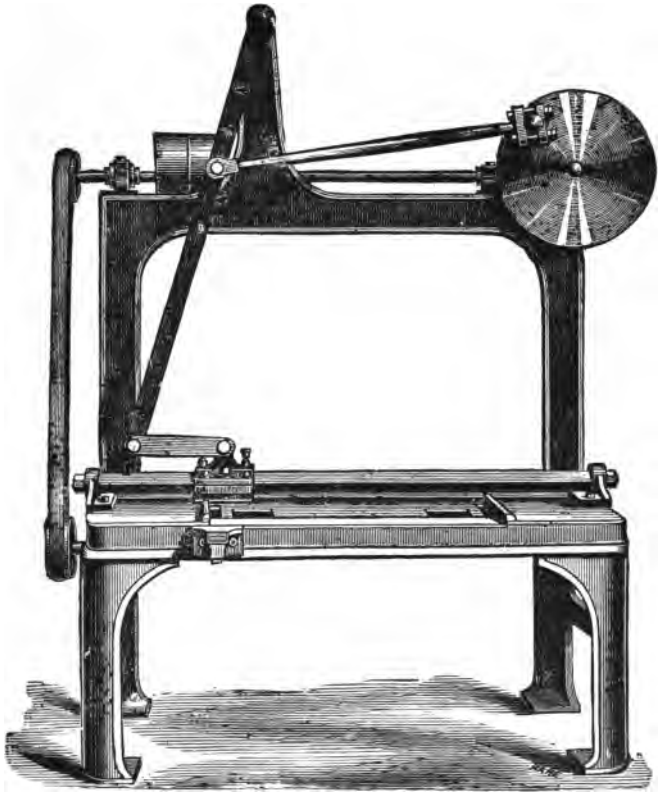

FIG. 5.—Cake Paring Machine.

without disturbing the driving wheels. The vertical shaft is made of cast-iron, about 7½ inches in diameter, having an oblong chamber or naval formed in the centre, into which is fitted a strong cast bush having a brass bush at each end through which the stone spindle passes. The bush is free to rise and fall in the naval as material is added to or taken from the stones. All the sweepers and sweeper frames are made of wrought iron. The circular pan is made of cast-iron, dished slightly, and having a rim round its circumference 7 inches deep. All the bearings are brass, made very heavy and

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unusually long. The ground parings are carried from the edge runners by a conveyer, from which they fall into the seed kettle. The process of refining the crude linseed oil is as follows. The crude oil is taken from the store tanks by a force pump into a lead-lined cistern, where it is mixed with the necessary chemicals from another cistern. After these have done their work and the process, as described hereafter,

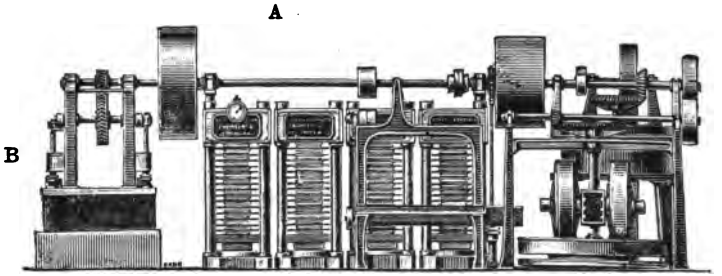

FIG. 6.—Pumps, Presses, Paring Machine, Edge Runners and Gearing.

completed, the oil is run into a third tank, where it is agitated by air from an air-pump to complete the bleaching and refining process.

Fig. 7 shows a complete set of the plant above described with the exception of the oil-refinery, constituting what is known as the Anglo-American Oil Mill, No. 4. It forms the standard size or unit of this machinery; larger mills are made up of multiples of this mill, and contain from eight presses upwards. The Anglo-American machinery,

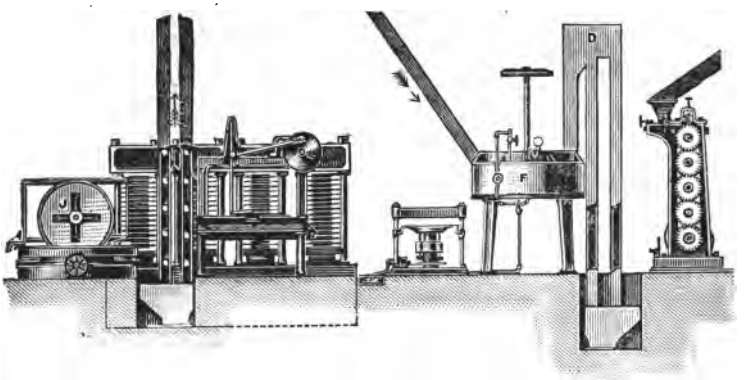

FIG. 7.—Unit Oil Mill.

as manufactured by Messrs. Rose, Downs & Thompson, is worked in sets of four presses as shown, and a mill can contain any number of sets. One set occupies a space 36 feet by 28 feet by 16 feet high, and takes 45 horse-power to work it. Additional sets will take about 35 horse-power each. *Résumé.*—The complete process is therefore as follows. The seed passes through rolls, C, from these an elevator, D, places it in the kettle, F, from this it passes to the moulding machine, G,

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where it is measured, formed and compressed, and then passed to the presses, H. The cakes, after being pressed, are cut to size in the paring machines and the parings reduced to meal by the small edge-stones, J. The parings are then returned by an elevator to the kettle, F, to be re-worked. The presses stand in a tank, into which the expressed oil falls and from which it is forced to the store tanks or filters by a pump. Fig. 8 shows an illustration of filter press.

Extraction of Linseed Oil from the Seed by Solvents.—As linseed does not lend itself kindly to extraction by solvents, the process need not be discussed here; those interested will find full particulars in Andés' *Vegetable Oils* (Scott, Greenwood & Co.). Moreover, even if it were more practicable to extract the oil from linseed in this way than it is, it is difficult to sell a meal devoid of oil.

The Chemical and Botanical Control of Oil Crushing.—Each de-

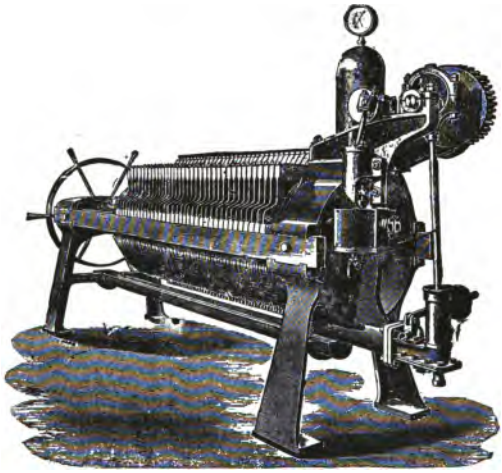

FIG. 8.—Filter Press for Oil.

livery of linseed should be carefully sampled, and the percentage of oil determined by means of apparatus shown in Figs. 9 and 10. Frequent determinations of the cake from the different presses should be made daily, so as to avoid making an unsaleable cake, on the one hand, owing to its low percentage of oil, or, on the other hand, leaving too much oil in the cake above the guarantee at which the oil miller sells his cake. *Example:* If an oil miller purchases 1,000 tons of linseed containing 35 per cent. of oil and he guarantees his cake to contain, say, 13·3 per cent. of oil, when it comes to stocktaking he should be able to account for 250 tons of oil and 750 tons of cake of above analysis.

Moreover, the oil miller should, if practicable, contract for his seedto contain a minimum percentage of foreign seed, and if he is not able to make a microscopic determination himself he should send a sample to the botanist of one of the national Agricultural Societies,

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say, the Royal Agricultural Society of England or the Highland and Agricultural Society of Scotland, so as to get the percentage of seed other than linseed. Should the gravity of the oil prove abnormal, then this botanical examination may throw light upon the matter. The botanist might also be asked to report on the presence or absence of weeds or their seeds, or vegetable organisms of any nature injurious to cattle, as there have been cases decided in court holding the crusher liable for the value of cattle dying through eating noxious cake.

FIG. 9.—Oil Extractor.[1]

FIG. 10.—Battery of Oil Extractors being Heated on Water-bath.

Methods of Storing.—Galvanised iron reservoirs are to be preferred for storing oil. Their base should be very wide in proportion to their height; they are generally covered by a badly fitting lid, so that the air may be constantly renewed on the surface of the oil, but it is preferable to use a tight-fitting lid so as to exclude dust. In this case a current of air is set up by two tubes placed on opposite sides of the reservoir, thus starting oxidation, and thereby increasing the drying properties of the oil. It is also of importance to maintain the oil at a constant temperature, about 15° to 20° C. It is advisable

[1] See note, p. 21.

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to draw off the "foots" two or three times a year. It is an undeniable fact that oil kept for a certain time gives a better coating on drying to that yielded by an oil fresh from the press. Speaking generally, linseed oil should not be used until after it has been "aged" for a year or two. Such oil is known as old tanked oil, and gives better results in varnish-making than can be obtained by any mere rapid process of bleaching freshly pressed raw oil.

Oil Refining.

Purification and Clarification.—The oil obtained from oleaginous seeds by pressure is very impure, and owing to the presence of easily decomposable foreign substances, amongst which albuminoid principles predominate, it cannot be used in its raw, unrefined condition. It must therefore be refined. *Mechanical Purification of Oil by Filters.*—The *first* stage of the refining process is *mechanical*, the *second, chemical.* The oil is purified *mechanically* by conveying it as it comes from the presses into a reservoir, from whence a pump distributes it to the filter presses, in which the greater part of the foreign substances which it holds in suspension are separated. *Chemical Purification—Refining.—Chemical* purification consists in treating the oil, *after it has been previously mechanically clarified,* in a methodical manner with dehydrating agents such as concentrated sulphuric acid.

By Acids: Thenard's Process.—By using a quantity of not too strong acid (1 to 1½ per cent. of 66° B.; sp. gr. 1·84), the acid exerts its action on the foreign matters contained in the oil in preference to the oil itself—it acts at first by absorbing moisture; it then attacks the foreign matters and transforms them into a carbonaceous mass, which imparts to the oil a brown coloration, which, according to Livache, quickly deposits as a flocculent precipitate. When the acid has completely charred these substances water is turned on to dilute the acid, so as to prevent it acting on the oil. After being energetically agitated with injection of air, and repeated washing with water heated by an open steam pipe, the oil is conveyed into large tanks and left to settle. It only remains to separate the purified oil by decantation. According to Hartley, however, oil treated in this manner is itself frequently charred, or some impurity therein which dissolving in the oil imparts thereto a brown coloration, which is not removed by the subsequent bleaching process to which the natural colouring principles of the oil are amenable. This brown colour in his experiments sometimes separated as a flocculent precipitate, but only after prolonged subsidence. Success in this process, in fact, depends upon adding just enough acid of the proper strength and no more as will be sufficient to char the mucilage without attacking the oil itself. Hartley states that he succeeded well with acid of 30 per cent. strength (!), which, whilst charring the mucilage, did not attack the oil when left in contact with it.

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[It must be borne in mind that however expert a chemist may be, that fact alone does not constitute him an oil refiner, and, as a matter of fact, linseed oil can be refined and bleached almost water-white by sulphuric acid alone by so-called ignorant workmen who have the advantage of being practical.]

This oil is not, however, free from acid. Linseed oil containing even a small proportion of sulphuric acid when heated to the high temperature incidental to varnish-making would char. This, of course, would be obviated by the use in such a case of a basic drier, such as litharge or manganese, but then only if kept sufficiently long in contact with every particle of oil by agitation. Metallic salts decomposable by sulphuric acid, such as sugar of lead, would act similarly. A very practical method of rapid filtration was given by Ure. The filter is composed of three parts, of which the filtering material, charcoal, waste, etc., occupies the middle. The oil, placed in an adjacent reservoir, communicates on one hand with the lower compartment, and on the other hand by a pipe leading from its bottom with a reservoir of water placed at a sufficient elevation. The water displaces the oil, and causes it to pass through the filtering material, and to be transferred into the upper compartment.

[In these days of filter presses, and working on the large scale, trivial filtering processes like the last two are rather out of date.] Those who wish to filter linseed oil on the small scale cannot do better than adopt the method used in sugar refineries, where Taylor's filter bags are employed—a wide bag enclosed in a narrow one acting as a compressing sheath. The apparatus may be enclosed in a lead-lined case. Other processes of purifying oils chemically have been recommended, such as a concentrated solution of chloride of zinc, as suggested by Wagner and recommended by Hartley, heating the oil with freshly calcined magnesia; coagulation of the albuminous matter by warm steam pipes, or by the injection of air heated to 110° C.; addition of tannin extract or of salts of iron or alumina to precipitate albuminous matter; partial saponification with a small quantity of caustic lye, so as to carry down the foreign matters with the resultant soap in much the same manner as beer is refined with isinglass, a process employed with success in the purification of cotton-seed oil. Hartley recommends a concentrated solution of sulphate of manganese. It would be superfluous to dwell longer upon these different processes. This short enumeration will be sufficient. The purified oil is then stored in large reservoirs.

Bleaching of Oil.—For high-class varnishes it is necessary to use a very pale oil. Even with an oil which only shows a very faint yellow tint the quality of the varnish into which it enters will be deteriorated. It is therefore necessary to bleach the oil. The colouring principles of oils are derived from powerful colouring matters existing in the fruits and seeds from whence they have been extracted. They may be resolved into four principal substances of very similar chemical composition: Xanthophyll and chlorophyll,

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both yellow, another, but blue, chlorophyll, and finally erythrophyll, of a red colour. These substances are not necessarily found in *all* oils, but nevertheless they are always present in linseed oil, and the variations in their relative proportions are the cause of the different colours which different samples of this oil exhibit.

If erythrophyll and a mixture of the two chlorophylls predominate we get a brown oil; if a greater proportion of chlorophyll be present the oil will have a greenish-brown tint; finally, if xanthophyll predominates a pale yellow oil is the result. These substances are decolorised by sunlight, especially in contact with air; oxidation also destroys them; they are also easily decolorised by dilute acids. Alkalies and certain metallic salts first precipitate the chlorophyll, then in the long run, if added in excess, the other two substances are likewise precipitated; finally, they are rapidly decolorised by chlorine and hypochlorites.

Five processes of bleaching oils are in use based upon the preceding remarks: (1) By sunlight alone, or by sunlight and air acting together; (2) by oxidising agents; (3) by acids; (4) by alkalies or metallic salts; (5) by chlorine.

1. *Action of Light.*—In decolorising oil the best result is obtained by the prolonged action of sunlight, and a superior article is said to be obtained to that yielded by the use of chemical reagents. This may be true of many chemicals, but not of oil refined by sulphuric acid. When linseed oil is exposed in a very thin layer to the direct action of sunlight, it bleaches in two hours. Working with large quantities the process is of course of longer duration, but by using large flasks of colourless glass, says Livache, and exposing the oil in these to direct sunlight, bleaching proceeds very rapidly, and so that the oxygen of the air may aid in the operation the mouths of the flasks are simply plugged with cotton wool. When it is desired to treat larger quantities the oil is placed in flat lead-lined or zinc-lined boxes about 40 inches long by 20 inches wide and 6 to 10 inches deep, covered with a glass plate, and slightly inclined by raising one of the sides of the box about ½ inch or so; or the glass plate is made to overlap the sides of the box, so that no rain-water gains access to the contents of the boxes, which are generally placed in the open air. Finally, two tubes lead into the box from the two opposite sides, so that the air on the surface of the oil is being constantly renewed. Linseed oil, it is said, can thus be bleached to a bright colourless oil in less than a fortnight.

Mulder previously filtered the oil through animal charcoal. Some expose the oil to sunlight in contact with animal charcoal for a week and do not filter the oil until then. But neither glass flasks nor even lead-lined boxes, nor, in fact, sun bleaching, whether aided by animal charcoal, etc., or not, in any way coincide with the practical notions of the present day. Sun-bleaching of oils is only to be found in books, and is so long obsolete that any who attempted it on the large

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scale would nowadays be legitimately regarded as crazy. Undoubtedly oil can be bleached almost water-white by the sun when either sunlight or daylight can get at it in every direction, but the interest on capital more than swallows up the profit, and the market for such an article is necessarily confined to artists.

2. *By Oxidising Agents:* (*a*) *Peroxide of hydrogen*, which is now easily obtainable in commerce, has also been recommended. The oil is shaken with 5 to 10 per cent. of peroxide of hydrogen of 10 per cent. strength. This process would appear to be only adaptable to small quantities. Expense would debar its use on the large scale, and, as a matter of fact, quite irrespective of expense, both peroxides of hydrogen and sodium have no practical value as oil-bleaching agents, however energetic they ought to be theoretically in this respect, and it is only a waste of time, money and patience to attempt to bleach oil in this way.

(*b*) *Ozone.*—Attempts have been made to use ozone. It is made by causing a current of air to pass through a series of ordinary ozonising tubes, where it becomes richer and richer in ozone, being finally led to a receiver containing the oil to be oxidised. The oil is heated by a steam coil to about 40° to 50° C., and the ozonised air is admitted to the bottom of the vessel, and is made to pass through the whole of the oil by means of a tube pierced with very small holes.

Schrader and Dumcke found that ozone only acts upon the oil for a comparatively short time—it in fact stops very quickly. But if the oil thus treated be placed in white glass flasks in flat boxes, in contact with air and preferably in the sun, the action continues of its own accord, bleaching and thickening the oil in a very short time, and causing it to dry much more rapidly.

Although information and particulars in regard to the bleaching of oils by ozone are far from definite, this process, says Livache, ought to receive the attention which it undoubtedly deserves. But so long as there are so many adventurers in the field, glib of tongue and adepts in persuasion, who simply live by imposing on those in the trade desirous of testing new methods in a rational manner, so long will such methods as bleaching by ozone be highly discounted. Each adventurer is more anxious than another to trap the unwary manufacturer. It is not the adventurer's wish, even if he were capable of bringing any such process to a successful issue, that it should succeed. His living would then be gone. His policy, before the final grand test which is to show such grand results, is to goad his patron on to a quarrel, as a result of which he leaves to trap some other unwary manufacturer, a rival in trade of the one he has just left, and who is only too ready to listen to any story of the expert's (*sic*) unjust treatment at the hands of his rival, and so he proceeds from factory to factory in his successful confidence-trick method of exploiting his process, and so the game goes on until it is naturally played out, when he finds some other fortune in less-than-no-time-making process to exploit in the same way, unless, perchance, in the

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interval he has not come to an untimely end in some more than usually rash and ill-advised experiment.

(*c*) *By Permanganate or Bichromate of Potash.*—The permanganate, or, better still, the bichromate of potash, in conjunction with sulphuric acid, have long been used as bleaching agents. The process is conducted in wooden tanks lined with lead. For every 100 lb. of oil about ½ lb. of bichromate of potash is mixed with double its weight of sulphuric acid, previously diluted with five times its bulk of water. This mixture is run into the oil in a thin stream, with constant stirring, which is kept up for an hour, the oil, if need be, being heated all the time by a steam coil. The liquids are allowed to separate, the lower layer drawn off, and the oil repeatedly washed with hot water. This process is tedious, owing to the difficulty of eliminating even by acid the green hydrated oxide of chromium which dissolves in the oil. The same remark applies to bleaching with manganese compounds where a similar hitch occurs. There is a great loss of oil with both processes altogether incompatible with the extent of any to which the oil may be bleached. However, in both cases the oil may increase in drying properties owing to the action of the nascent oxygen developed in the processes in question. *By Linoleate of Manganese.*—Blenkinsop and Hartley (see p. 32) proposed to bleach linseed oil, or rather to produce a "boiled" oil paler than "raw" oil, by oxide of manganese introduced in the state of linoleate of manganese dissolved in coal-tar naphtha. The process involves the use of heat. The oxygen used up in oxidising the oil and bleaching the colouring matters is restored to the manganese by a current of air as fast as it is deprived of it. But this process was well known both in Britain and America long before the date of this patent.

3. *By Acids:* (*a*) *By Nitric Acid, etc.*—Lawson has suggested dilute nitric acid. The process should be most carefully watched, so as to prevent any elevation of temperature. In England, according to Livache, use is made of a mixture of nitric acid and chlorate of potash in the proportion of 1 to 2 per cent. of the oil to be decolorised; heat is applied, and the oil is then repeatedly washed with water. This process, he says, has been successfully employed with cotton-seed oil, but as a matter of fact the caustic soda method for refining cotton-seed oil and the sulphuric acid method for refining linseed reign supreme in Britain, and this is in accordance with the fact that it is principally coloured *mucilaginous* impurities which have to be got rid of in refining linseed oil, whilst it is coloured *resinous* impurities which are present in crude cotton-seed oil.

(*b*) *Sulphuric Acid*, see pp. 15-16.

4. *By Alkalies and Metallic Salts:* (*a*) *By Caustic Soda.*—Certain oils, containing resinous colouring principles, particularly cotton-seed oil, may be completely decolorised by agitation with a small quantity of *caustic potash* or *soda* (1 per cent.). Combination with the colouring principles ensues, and as a result these are precipitated as cotton-seed oil foots, from which the purified oil may be easily run off

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through a syphon or otherwise. It is not applicable to linseed oil—at least to the same extent.

(*b*) *By Carbonate of Potash.*—5 lb. of the carbonate dissolved in 10 gallons of water are added to 100 lb. of oil, and the mixture stirred to thorough incorporation; 2½ gallons of a 2 per cent. solution of chloride of calcium are then added. The oil bleaches rapidly, and it is then decanted and treated with a 5 per cent. solution of sulphuric acid, and afterwards washed until perfectly neutral. But this would be far too laborious and expensive a process for adoption on the large scale, and like others has nothing to recommend it over the sulphuric acid process.

(*c*) *By Ferrous Sulphate: Artists' Oil.*—Amongst the salts proposed, ferrous sulphate (green vitriol) is capable of giving good results, with small quantities of oil. *Process.*—One part of green vitriol is dissolved in 1½ parts of water, and this mixture is added to double its volume of linseed oil contained in a glass flask. The whole is then exposed to sunlight, and shaken at least once a day. The oil is generally bleached in from three to six weeks, according to the amount of sunshine. When the oil is decanted the green vitriol solution can be used over again for treating a fresh quantity of oil. This process is much in vogue with artists' colourmen (the bleached oil being sold as *artists'* oil), but is too tedious, laborious and costly for adoption on the large scale.

(*d*) *By Basic Acetate of Lead.*—A solution of basic acetate of lead when agitated with oil eliminates its colouring principles very effectually.

(*e*) *By Sulphate of Lead.*—This process also gives good results. It is mixed with the oil to the consistency of cream, and exposed as in the preceding process to sunlight, with frequent agitation. After a time the oil is bleached. Two layers are found at the bottom of the flask, one consisting of sulphate of lead, the other of colouring matter. The sulphate of lead may be used over again. The rationale of the process is obscure. It may to a certain extent be looked upon as similar to the clarification of beer by isinglass.

(*f*) *By Complex Mixtures.*—More or less complex mixtures of different metallic salts are also used. We will only name the following, which gives good results, without seeking to demonstrate the precise reactions which simultaneously occur. To 10 gallons of linseed oil are added 1½ gallons of water, containing ½ lb. of black oxide of manganese, ¼ lb. of bichromate of potash, ¼ lb. of carbonate of soda and ¼ lb. of common salt. The boiling solution is added to the oil; it is left to clarify, and the colourless oil is decanted.

5. *By Chlorine.*—Substances from which chlorine can be generated without the aid of heat are added to the oil. For instance, 5 lb. of concentrated muriatic acid, 33 per cent., diluted with four times its weight of water, are added to 10 gallons of oil. The whole is well stirred, whilst a solution of 1 lb. of bichromate of potash in 1 gallon of water is added. A mixture of red lead and hydrochloric

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acid may also be used. To 10 gallons of oil ½ lb. of red lead beaten up with ½ lb. of oil is added, and the whole well stirred, whilst ½ lb. of hydrochloric acid is added. During the next five days an additional ½ lb. of hydrochloric acid is added daily, the whole being well stirred several times during the twenty-four hours. The decanted oil is run into large bottles, or placed in boxes lined with lead, and exposed to sunlight until the oil is perfectly colourless.

Summary.—The most rapid method of bleaching oils, according to Livache, is by means of chemical reagents, especially chlorine, but it must not be forgotten, he adds, that it is difficult to free the oil from all trace of these reagents, which in the end may exert a vexatious and injurious influence upon the final products manufactured from an oil bleached in this way. As a matter of fact, chlorine does not bleach linseed oil, it darkens it. Certain bleaching agents, like chlorine and nitrous acid, are apt to form substitution compounds which alter altogether the nature of the oil, and not for the better. Had one space at his disposal, and a sufficient supply of refined oil to meet his wants in the meantime, says Livache, the bleaching of oil by means of sunlight in flat boxes covered with glass plates cannot be too highly recommended. The slowness of the process would, he says, be largely compensated by the beauty and quality of the products manufactured from such an oil.

However that may be, an equally good product can be obtained by bleaching with sulphuric acid in fewer hours than it takes weeks by sunlight. In fact, bleaching by sunlight could only be recommended by those who have never seen oil refined by sulphuric acid, which is the most effectual, the most expeditious and the most economical process, requiring no costly plant, no costly chemicals, only about 1 lb. of oil of vitriol per 10 gallons of oil. A steam pipe heated by exhaust steam mixes the acid and oil together, and does all the agitation necessary for washing the acid out of the oil, all the operations being done in the one tank if need be. No process could be cheaper, no process could be more simple, but it requires care and experience, as it is quite easy to irretrievably spoil 5 to 10 tons of oil which can then only be used for boiled oil.

NOTE.—*Explanation of Fig.* 9.—The solvent distilled from B ascends right tube of A into condenser, K, from which it falls on to weighed amount of material to be extracted in the wide central tube of A, from which, charged with oil, it runs off automatically through left syphon tube into B, whence it is redistilled, and so the cycle goes on until the material is exhausted of oil, when the flask is detached, the ether distilled off, and the residual oil weighed.

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CHAPTER II.

OIL BOILING—THEORETICAL AND PRACTICAL.

Chemical Constitution of Drying Oils.

Linolein.—Since all oils consist of carbon, hydrogen and oxygen in almost identical proportions, it may well be asked what principle it is that is present in certain oils and causes them to dry, and which must therefore be absent in those oils which do not dry. Mulder found that the principal constituent of linseed, poppy-seed and walnut oils—as well as that of all drying oils—was *linolein*, a substance formed by the combination of linoleic acid with glycerine. His conclusions have been partially confirmed by Hazura and Friedreich, who found, in poppy-seed and walnut oils, fatty acids analogous to, if not identical with, linoleic acid. More recently, however, as a result of fresh experiments, Hazura has been led to regard linoleic acid as composed of two distinct acids, *linolic* and *linolenic*. But Reformabsky disputes these conclusions. As a consequence of still more recent researches by Livache (*Comptes Rendus de l'Académie de Science*, 1895), he holds that the theory that the drying properties of linseed oil are entirely due to the presence of linolein can hardly be maintained. In fact all other oils, whether of vegetable or animal origin, are capable of being transformed into a solid product analogous to that to which linseed oil is so easily converted, and that whether taken individually or as the component parts of a mixture, provided always that they be submitted to the action of heat. But actual facts are against any such conclusions; no amount of heating or treating with driers will ever make castor oil "dry". It is true the oil may be converted into a plastic solid by heat and driers, but, however useful such a substance might be as a pitch substitute, it is of no value as a drying oil; non-drying oils rancidify but they do not dry. According to the present state of our knowledge of this subject, continues Livache, it seems that the best explanation that can be given of the drying properties of oils is the following: *All* the different glycerides which enter into the composition of a drying oil play a part in the transformation of the oil into a solid body, but the greater the proportion of one or several glycerides analogous to linolein the more rapidly is this transformation effected, and at a

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lower temperature. All the glycerides present in oils and fats undergo this change more or less slowly, as if by a kind of metamorphosis. Neither can this doctrine be upheld. It is ridiculous to say that the stearine of cotton-seed oil plays any part in drying but a negative one. Looking at the matter from this standpoint, we are able to explain the differences—in drying properties—of different drying oils. Should in fact the glycerides, analogous to linolein, be present but in small quantity, we can understand that the other glycerides will only dry slowly, and very often we must resort to the aid of heat to accomplish the object in view. Whatever explanation we adopt, we must bear in mind that the drying oils are those which may be quickly converted into a solid elastic substance at the ordinary temperature. This transformation can only take place in the presence of oxygen, and with a rapidity which varies much according to the heat and light the oil is exposed to, and the previous treatment to which it has been subjected.

Linoxin.—In the case of linseed oil Mulder called the resulting solid product *linoxin*. He found that it did not differ from linoleic acid but by containing a larger proportion of oxygen, whilst at the same time all the glycerine had disappeared. While making reservations necessitated by the still imperfect state of our knowledge regarding the composition of the different solid bodies obtained by the oxidation of different oils, we shall retain the name of linoxin, for this body, no matter what oil it may be derived from, presents the same properties of elasticity, insolubility in the usual solvents, etc., etc. Linoxin consists of a perfectly dry elastic mass, of a more or less deep yellow or brown colour according to the treatment the oil from which it has been derived has been subjected. For a long time it was believed to be perfectly insoluble in the different menstrua in which oils dissolve. When exposed to their action it at first undergoes no change, but if the action be prolonged it increases in transparency, swells like indiarubber, and at the same time a small proportion dissolves. The resulting swollen substance dried apart from the solvent is still elastic, but very friable between the fingers, crumbling to particles, with no tendency to reunite. By evaporating the solvent there is left a tacky residue of low melting-point. The oxidation product of a drying oil, therefore, presents many analogies to caoutchouc, being composed like it of two constituents, one of which dissolves in different menstrua, whilst the other swells and disintegrates. The dissolved product recovered on evaporation of the liquid solvents acts as a real cement, reuniting the insoluble portions, first swollen and then disintegrated, yielding as a result a continuous, elastic mass, consisting on the one hand of the soluble, and on the other hand of the insoluble, portion of the original linoxin. But very possibly this liquid portion found by Livache was simply incompletely oxidised oil. Thus before an oil can dry, the linolein must be in a position to become oxidised, and the more this is facilitated the quicker does the oil dry, as the oxidation of the other

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glycerides would appear to be one of the functions of the oxidation of the linolein.

Oxygen may be caused to act either upon the linolein, *i.e.*, upon linoleic acid combined with glycerine, or upon the linoleic acid separated from glycerine, or finally upon suitable chemical combinations of linoleic acid with metallic oxides, *i.e.*, upon linoleates.

Action of Oxygen upon Linolein.—Linseed oil exposed to the air in a thin layer soon changes to a solid substance. If we perform the same experiment in a vessel containing air placed mouth downwards over mercury, the same change takes place, but the volume of air confined over the mercury diminishes in volume owing to the absorption of oxygen. Finally, if the quantity of air suffices, the oil is converted into a solid product which has increased in weight proportionally with the oxygen which existed in the air and which has disappeared, whilst the residual gas is composed of nitrogen, together with a small quantity of carbonic acid, and volatile acids of the methane series. The transformation of the oil and consequently of the linolein is thus due to the action of oxygen. Mulder obtained this solid body by exposing the oil on plates to the action of the air, and after detaching, he treated the product with ether, alcohol and water so as to wash away any unoxidised oil or other soluble matter; and he finally obtained a more or less elastic white substance which analysis showed to be a product of the oxidation of the anhydride of linoleic acid, *viz.*, linoxin. From linoleic acid exposed in a thin layer to the air he obtained the same solid linoxin, but the change into a perfectly dry substance took longer than in the case of linolein. But the products are identical in composition and properties, and as no glycerine is found in the product of the oxidation of linolein by the air, it would appear that the oxygen of the air first acts upon the glycerine, yielding such bodies as carbonic acid, water, etc., which disappear, and then upon the linoleic acid of the linolein, converting it into a solid body, linoxin. The product is identical whether we use air or oxygen. *Effect of* (1) *Heat and* (2) *Light and* (3) *Different Rays on Rapidity of Drying.*—(1) Heat exercises a very decided influence; oxidation takes place more rapidly, whilst at the same time, the oil being more fluid, the oxidation is more thorough owing to the oxygen penetrating the oil better and more deeply. Every one knows that paint and varnish dry better in summer than in winter. (2) The *intensity of the sun's rays* has a very decided action. Oil exposed to direct sunlight dried in four days, whilst the same oil in similar circumstances, but in semi-darkness, took fifteen days to dry. (3) According to Cloez, *when the light is caused to pass through coloured glass the oxidation varies with the colour.* The maximum amount of oxidation takes place with colourless glass, but with blue, red, green, or yellow, a longer time is required the nearer the colour approaches to yellow. (4) *When oil is exposed to air in darkness*, it is longer before the oxidation process sets in, and it takes a very long time for complete oxidation.

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Action of Oxygen upon Linoleates.—Linolein is very easily saponified, yielding soaps; potash, soda and ammonia yield soaps which readily dissolve in water. Baryta, lime, the oxides of zinc, copper and lead, yield soaps insoluble in water but soluble in ether. The most suitable combination to study is that of linoleic acid with oxide of lead; if we dissolve this linoleate of lead in ether, and if we expose the liquid in a thin layer upon a plate of glass, the white solid residue remaining on evaporation of the ether, which is at first soft, becomes in a few days very hard, owing to the absorption of oxygen.

Linoxic Acid.—If we suspend this hard, brittle salt of lead in alcohol, and pass a current of sulphuretted hydrogen through the alcohol, we obtain, after filtering off the sulphide of lead, an alcoholic solution from which water precipitates a white substance, which analysis shows to be that oxidation product of linoleic acid to which the name of linoxic acid has been given. If instead of separating this alcohol in the cold by the simple addition of water, we evaporate the alcoholic solution by the aid of heat, we also obtain a viscous residue, but of a blood-red tint. Linoxic acid is therefore met with in two colours—white or red—according as it has been prepared in the cold or the hot state, *i.e.*, whether hydrated or anhydrous. But whilst viscous linoxic acid exposed to the air changes to dry elastic linoxin, on the contrary, when combined with lead—although it also suffers this alteration—it becomes more and more friable.

Résumé.—(1) *Linoleic acid* combined with glycerine, in the state of *linolein*, yields progressively in a more or less short period of time *linoxin*, a solid elastic body, a basis for colours and varnishes. (2) Free linoleic acid yields fairly quickly a viscous compound (linoxic acid) which afterwards changes to linoxin, but after a longer period of time than in the preceding case. (3) Linoleic acid combined with oxide of lead, *i.e.*, *linoleate* of lead, dries fairly rapidly in consequence of the formation of *linoxate* of lead, but this product changes afterwards into a friable, brittle substance. It follows that, to ensure a dry, elastic product, we ought preferably to cause the oxygen to act upon the linolein and to avoid, as far as possible, either the presence of linoleic acid, which would take a longer time to dry, remaining viscous for rather a long time in consequence of the formation of linoxic acid, or an excess of linoleate of lead, which would give a brittle, friable product. Owing to the difficulty of separating linolein from the other principles entering into the composition of oils in actual practice, we have to oxidise the oil itself. The drying properties of a drying oil are increased under certain conditions, which it is important to study in detail. We shall study, therefore, how the drying properties of linseed oil—the best drying oil—may be increased, for whatever we may determine regarding it holds good, keeping to the same proportions with other drying oils.

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Methods of Increasing the Drying Properties of a Drying Oil.—The rapidity of drying is influenced by: (1) The degree or extent to which the oil has been refined; (2) the time which has elapsed since its extraction; (3) the conditions under which it has been preserved; (4) heat; (5) addition of certain substances.

1. *Extent to which Oil has been Refined.*—The oil as it issues from the press contains water and impurities, *mucilage* "*foots*". It is evident that to obtain a continuous elastic mass, it would be advantageous to get rid of these substances by allowing the oil to clarify itself by simple deposition, by filtration, or by chemical treatment. In the latter case, the oil is generally treated with sulphuric acid, which frees it from substances which play no part in the drying of the oil, because they do not fix oxygen; if we expose to the air comparatively freshly pressed oil, and the same oil, after treatment with sulphuric acid, then freed by washing with hot water from all trace of acid, we find that the oil in the second case absorbs a larger quantity of oxygen. Moreover, the paler the oil the whiter will be the final product, *i.e.*, the dried coat of linseed oil thus demonstrating that the oil ought to be bleached before application.

2. *Age of the Oil.*—Oil preserved from contact with the air absorbs oxygen the more rapidly the longer it has been stored, owing possibly to a polymerisation of certain principles of the oil. Fahrion thinks that the non-saturated fatty acids enter into combination with themselves to form complex addition products.

3. *Method of Storing the Oil.*—Leaving age out of account, if the oil has been stored in contact with air it will already have absorbed a certain quantity of oxygen. Now experience shows that if, in the beginning, the oil absorbs oxygen rather slowly, this absorption, once fairly started, goes on afterwards much more rapidly. In the case of such an oil the absorption of oxygen will take place much more rapidly when applied to any surface exposed to the air.

4. *Action of Heat.*—The temperature at which an oil is exposed to the air has an influence on the rapidity of drying of that oil. Linseed oil dries more rapidly at a temperature of 25° to 28° C. than at a temperature of from 15° to 18° C. (Chevreul). Further, if we expose raw linseed oil under the same conditions of temperature to the action of air, and the same oil, after having been previously submitted to the action of heat, the drying property is altered. If we heat linseed oil for three hours, so that it only disengages a few gaseous bubbles from time to time, the drying properties of the oil are greatly increased; as has been shown by Chevreul, it takes only about half the time to dry. Fahrion states that when boiled in a deep pan, so that oxygen of the air cannot intervene to any great extent, the polymerisation of the non-saturated fatty acids is accelerated with the formation of complex substances which absorb oxygen more rapidly; a very unreliable statement. But Chevreul found by prolonging the heating of the oil—the temperature, etc., remaining constant—that oil heated for five hours took longer to dry than oil heated

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for only three hours. It is probable that under the influence of prolonged heating a certain proportion of the glycerine is destroyed (as the smell of acrolein would appear to indicate) with the simultaneous liberation of linoleic acid. We have seen that linoleic acid takes longer to dry than linolein, which probably explains why Mulder found that oil heated to between 70° and 100° C. took longer to dry than raw oil. What we can say is that: (1) A regulated temperature stimulates the drying properties of raw oil; (2) exposed to the same temperature raw oil dries less quickly than when it has been previously submitted to the action of heat, provided always that the heat is regulated so that the oil does not decompose in such a way as to liberate free linoleic acid.

5. *Addition of Certain Substances: Driers.*—White lead, litharge, black oxide of manganese, etc., stimulate the drying properties of a drying oil. Sometimes these substances may be added in the cold, sometimes the mixture is heated. The process of oil boiling in most general use is that in which the oil is simply heated with a lead compound. The increased drying properties of the oil have by some been attributed to the formation of linoleates of lead with the simultaneous production of free linoleic acid on heating, whilst others aver that the oil becomes oxidised at the expense of the linoleate of lead, as borne out by the fact that a certain proportion of metallic lead is found at the end of the operation. This double explanation is not satisfactory for the following reasons: because linoleate of lead only imparts a fictitious drying property it very soon becomes brittle and friable, whilst, on the other hand, linoleic acid dries less quickly than linoxin. It is, however, an incontestable fact that an oil heated with oxide of lead dries quicker than raw oil, or oil heated by itself alone. The question then is whether the oil increases in drying properties at the expense of the oxygen of the oxide of lead.

Again, good linseed oil exposed to the air absorbs 16 to 18 per cent. of its weight of oxygen before it dries completely, and on the other hand, as the quantity of oxide of lead, litharge or red lead does not exceed 3 to 8 per cent., at the highest estimate, of the oil to be boiled, it will be seen that the amount of oxygen which this oxide of lead could yield would be less than 1 per cent. instead of the 16 to 18 per cent. which is necessary, and consequently that it can only play a very secondary part as far as the direct oxidation of the oil is concerned, much more so as a part of this oxide does not yield up any of its oxygen to the oil. Yet a certain quantity of metallic lead left at the end of the operation proves that a corresponding quantity of oxygen has started the oxidising process, and we have seen, in studying the action of oxygen upon raw oil, that the drying properties of an oil increase more rapidly after the oil has reached a certain stage of oxidation. But the remarkable increase in drying propensities altogether out of proportion to the oxygen equivalent to the reduced lead proves that this reaction would in any case appear to be of a secondary nature. The same objections and the same explana-

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tions hold good in regard to the function of manganese compounds as driers.

It was for a long time imagined that during boiling the oil was oxidised and its glycerine decomposed, but analysis of boiled oil shows that it still contains almost the whole of its glycerine, and this is as it should be, for linoleic acid dries much more slowly than linolein. The theory of the driers acting as oxygen carriers is only very partially, if at all, true, as oxidation is shown by analysis not to take place to any considerable degree. Moreover, the iodine value of the original oil is not diminished to any great extent even when the oil has been treated by ozone. If the oil were oxidised to any extent the iodine values would be correspondingly lowered.

Influence of Surface on Rapidity of Drying.—But Chevreul gives a better explanation. He showed that if we spread linseed oil on lead foil, freed from all traces of oxide, the drying qualities of the oil increase greatly. The oil cannot in this case absorb any oxygen but what the air can in the normal state of the oil supply. The greater rapidity of drying can only therefore be attributed to the presence of the metal, leaving out of account the combination in which it may enter the oil. [But neither Chevreul nor Livache take into account the action of free fatty acid on metallic lead, so this experiment of Chevreul's proves nothing, but that the oil contained or developed free fatty acid, and that this free fatty acid attacked the lead, and that the oil dissolved the resulting nascent linoleate of lead, and thus hastened the oxidation process.]

But we may go further, continues Livache. If we shake raw oil with very porous metallic lead—obtained by precipitating a salt of lead by another metal—in a flask deprived of air, we find that, without applying any heat whatever, the oil dries much more quickly than before treatment, and in this case it is certain that there has not been any oxygen supplied to the oil. The only certain modification which the oil has undergone is the presence of a small proportion of lead, and we are thus led to conclude that the reason the oil dries quicker is due solely to the presence of this small quantity of lead. But here again Livache omits the one important point, the action of the fatty acids invariably present in the oil, and which will act much more rapidly on finely divided lead. In the case of manganese we cannot avail ourselves directly of this experiment, but by an artifice we can place the oil in the same conditions with regard to manganese. The oil is previously treated with the precipitated lead, then with a solution of a salt of manganese, the sulphate for example, which by double decomposition will yield sulphate of lead insoluble in the oil, whilst at the same time the manganese takes the place of the lead. It has thus been found that the resultant oil dries quicker, better even than that obtained by heating raw oil with a manganese salt. It is by studying the action of a manganese-treated oil that an explanation has been found of the cause of the increase in drying. If we spread such an oil in a thin layer in contact with air, it will

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assume a brown tint, due to the passage of the oxide of manganese present to a higher degree of oxidation owing to the absorption of oxygen from the air, but this brown coloration fades as the oil becomes more viscous, finally disappearing altogether. The oxygen of the peroxide of manganese is used up in oxidising the oil, and in the end a perfectly colourless solid mass is obtained.

This coloration, followed by complete decoloration, shows that the oxide of manganese has played the part of an intermediary or carrier of oxygen, becoming easily peroxidised, and afterwards giving up its excess of oxygen to the oil. The action of oxide of lead is probably similar but less energetic. We are, in fact, face to face with one of those phenomena, the inference to be drawn from which has been defined by M. Berthelot as follows: "We see by these phenomena how latent energy, and energy *capable in theory of producing exothermal phenomena*, but which under given circumstances *do not* produce such phenomena, may be rendered manifest by the intervention of certain agents acting solely as intermediates or go-betweens, and capable of developing reactions which may go on indefinitely. This is the foundation of the whole theory of thermochemistry as enunciated by me in 1865" ("Action by Presence," *Comptes Rendus de l'Académie de Science*, 1889, t. 109, p. 546). In a litharge or manganese-treated oil the lead or the manganese plays the *rôle* of intermediaries, taking the oxygen from the air and giving it up to the oil, which becomes oxidised more rapidly than without the aid of these intermediaries, and this reaction goes on to a certain extent during boiling, but when the oil is exposed to the air on a given surface it only ceases when the layer of oil has absorbed its maximum of oxygen. The catalytic action of the drier is not brought into full play until the oil has been exposed to the air in the form of the usual thin coat of paint or varnish. Vincent also defined this action as *catalytic*, but only as far as manganese was concerned. In virtue of the fact that both rosinate and linoleate of manganese dissolve in the oil, Vincent's hypothesis requires modification. Litharge and lead salts in his opinion dissolved in the oil, and thus intensified its drying properties, whilst manganese salts simply acted as carriers.

To find whether other metals could perform the same function as lead and manganese, a litharge-treated oil was stirred with a salt of the different metals to be tested, the acid of which would give with lead a salt insoluble in the oil. Livache found that litharge-treated oil spread on a glass plate dried in twenty-four hours, when manganese replaced the lead in six hours, whilst, when the lead was replaced by copper, zinc or cobalt, the oil took thirty to thirty-six hours to dry, and finally the oils in which lead was replaced by nickel, iron, chromium, etc., did not dry completely until after forty-eight hours. A very important point, says Livache, to be borne in mind is the fact that an oil indirectly treated with manganese by the substitution of manganese for the lead of a litharge-treated oil dries much faster than

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an oil directly treated with manganese. But it is very difficult to see why this should be so.

Lead and Manganese Salts.—Besides the *oxides* of lead and manganese we may employ certain *salts* of these oxides. But their choice would appear to be subordinate to their degree of solubility in the oil, and to the way they behave when heated along with the oil. Further, all the numerous salts proposed have been discarded except the acetate (sugar) of lead and borate of manganese, and the reason is not far to seek. These salts decompose when heated, and yield as a final result either the oxide of lead or finely divided metallic lead, or oxide of manganese, of which the valuable function is well known.

1. *Acetate of Lead.*—This salt melts in its water of crystallisation at about 75° C. Above 100° C. it loses water and a little acid, yielding the sesquibasic acetate, which towards 280° C. is completely decomposed, giving off carbonic acid and acetone and leaving as a residue metallic lead in an extremely fine state of division (spongy lead). We have seen the important *rôle* this plays in stimulating the drying properties of oil.

2. *Borate of Manganese.*—Again, on the other hand, we have in borate of manganese a very unstable salt, as is the case with borates in general. The affinity of boracic acid for oxide of manganese being but very feeble, the latter is liberated by the action of heat; the employment of this salt is therefore a useful roundabout way of introducing oxide of manganese into the oil.

As non-oxidised metallic lead, in a very porous condition, increased the drying properties of the oil, it was interesting to study the action upon oil of other metals capable of being easily precipitated. But the oil dried no quicker when treated with precipitated tin or copper. Besides, these results might have been foreseen, for Chevreul showed that linseed oil, spread upon well-polished lead, dried much more rapidly than when spread upon copper, brass, zinc or iron. With these metals the oil did not dry any faster than upon plates of glazed or unglazed porcelain, glass or plaster of Paris. As to the numerous other substances besides the salts of manganese and lead proposed to be added to drying oils to hasten their drying properties, none of them appear to exert any beneficial influence. Chevreul made a comparative study of the oxides of zinc and lead and showed that the former had no appreciable influence, and that certain substances even acted in a contrary manner, retarding instead of hastening the drying of the oil, such as the oxide of antimony and antimonious arseniate. If certain substances *appear* to act as driers, the reason is to be found in the fact that they have been used in conjunction with the application of heat, and their apparent beneficial action is due to the heat alone. According to Andés the following substances may be regarded as *absolutely useless:* all organic matters (sepia, dog excrement, bread, onions, garlic), red oxide of mercury, verdigris, lime, brass, zinc, alum, hydrated oxide of iron, boracic acid, oxide of antimony, gypsum, vermilion, pumice-stone, animal charcoal, and the following as *Oxidising*

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Agents.—White lead, sulphate of lead, carbonate of lead, basic acetate of lead, black oxide of manganese, hydrate of protoxide of manganese, sulphate of zinc, oxide of zinc, umber, and those in this final list as *Energetic Oxidising Agents.*—Air—acting through its oxygen—red lead, litharge and the different oxides of lead, borate of manganese and the hydrated peroxide of manganese.

Recently Kastner has proposed the plumbates of the alkaline earths as driers. These are prepared by heating in a suitable furnace two molecules of baryta, strontia or lime, or their corresponding carbonates with one molecule of oxide of lead. The plumbate of baryta is dense black, that of strontia, brown, and that of lime, bright red. Kastner is of opinion that the drying property of the oil is increased by the richness in oxygen and the introduction of lead; he holds further that the introduction of the alkaline earths themselves may produce oleates, which after drying assume a consistency of remarkable elasticity. *We ought therefore to confine ourselves in the use of driers to lead and manganese, to their oxides, and in certain cases to some one of their salts.*

Catalytic Action of Driers.—An experiment of Chevreul lends support to the theory of looking upon manganese and lead as simply performing the function of intermediaries or oxygen carriers, *viz.:* If we make a mixture of raw linseed oil and manganese-boiled oil, the liquid resulting from this mixture has a much greater oxygen absorbing power than either of the liquids constituting the mixtures taken separately.

If this be true, then a pale boiled oil made by diluting a manganese boiled oil with raw oil ought to be a very desirable mixture indeed.

It follows that the quantity of oxygen absorbed during the same period of time is not proportional to the quantity of oxide of manganese contained in the oil, since the boiled oil reduced with raw oil absorbs more oxygen, although bulk for bulk it contains less oxide of manganese than the original manganese-boiled oil. The only feasible explanation is this: If the raw oil in the mixture absorbs a greater quantity of oxygen in the same space of time, it is simply because this oxygen is supplied to it under more favourable conditions. Now these conditions are precisely the fixation of oxygen by the oxide of manganese, which forthwith gives it up to the oil; there is thus a continual transport of oxygen from the air to the manganese and thence to the oxidisable principles of the oil.

It seems, therefore, that an oil will dry in a better manner the less metallic linoleate it contains, because linoleate of lead absorbs less oxygen than linolein and becomes brittle; all we have to do is to introduce a quantity of lead or manganese sufficient to extract from the air the quantity of oxygen necessary to oxidise the linolein as fast as this oxidation proceeds. A large quantity of lead or manganese does not hasten the oxidation—the intensity of which is the sole function of the drying quality of the oil used—but would on the contrary yield a less elastic and slower forming final product. Again,

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summing up the proper conditions under which the drying properties of an oil are stimulated we can deduce the following principles: (1) The oil ought to be refined and clarified; (2) there is an advantage in allowing the oil to age before using it; if need be, oxidation may be started; (3) it is more preferable to use boiled oil than raw oil; (4) the drying of raw oil or boiled oil is hastened by the addition of either lead or manganese compounds in known quantities, or by metallic lead in a fine state of division. It remains to consider how in actual practice we can conform to the principles enunciated. In actual practice the oxides of lead and manganese or their salts are preferred; nevertheless the use of metallic lead has lately been tried with promising results. Driers are generally incorporated with the oil by aid of heat. However, interesting attempts have been made to effect this result in the cold.

Treatment of Linseed Oil with Driers in the Cold.

Numerous attempts have been made to prepare quick-drying oils without the intervention of heat, the great inducement being economy and a paler oil. Few processes have, however, been published. The simplest plan consists in passing the oil into a reservoir, from whence it falls upon plates superimposed at alternating inclinations and at certain distances one from another. The oil thus runs in a thin layer in a zigzag course from one plate to another and thickens as it absorbs oxygen; by using lead plates the action is more rapid. The oil when it reaches the bottom of the column of plates is pumped up to the top, and the same process is gone through again, and so on until the oil has assumed the requisite consistency. The theory of this is very simple, the oil oxidises, generates fatty acids which attack the lead and the resulting nascent linoleate of lead dissolves in the oil.

1. *Bink's Method.*—In Bink's process a very small quantity of drier (2 to 6 parts in the thousand), consisting of a mixture of oxide of manganese and a salt of lead is first added to the oil, then a current of air is passed through the mass until the desired quantity of oxygen is absorbed. But this process does not give satisfactory results without the aid of a moderate heat.

2. *Bouis' Process.*—Bouis proposed to introduce oleate of lead into the oil; the product is colourless and dries well enough. It has been proposed later to replace the oleate by the linoleate of lead, but the latter, it is said, does not give the same result as it is said to be insoluble in oil in the cold. Hartley and Blenkinsop dissolve the linoleate of manganese in naphtha and introduce it in solution in that vehicle into the oil and then blow air through it. But according to Professor Sabin, this process was well known in America long before Hartley, etc., patented it. Moreover the writer recommended it in his Polytechnic lectures several years before. See Vincent's method of oil boiling, p. 49.

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3. *Liebig's Method.*—Liebig recommended the agitation of the oil with water, litharge and basic acetate of lead. The latter salt is prepared by grinding as finely as possible 1 part of acetate of lead with 1 part of litharge to thorough incorporation, and placing the mixture in a porcelain basin, which is heated on the water bath and covered with another porcelain basin to prevent access of air. After an hour's heating a white mass is obtained, which is triturated with 5 parts of water. The solution on standing and clarifying contains the basic acetate of lead. It is diluted with its own bulk of water, and vigorously shaken with 20 parts of oil which has been triturated with 1 part of finely ground litharge. The mixture thus obtained is added to the oil to be treated, which it soon decolorises, and at the same time stimulates its drying properties. In those cases where the presence of lead would be prejudicial to the object for which the oil is to be used, it is agitated with a small quantity of sulphuric acid diluted with 3 parts water. The lead may also be eliminated by agitating the oil with a salt of manganese, the acid of which forms an insoluble lead salt—the sulphate, for example, or, better, the borate.[1]

4. *Livache's Process.*—Livache's process consists in agitating the oil in the cold with finely divided metallic lead, perfectly free from oxide. For this purpose he uses the spongy lead obtained by precipitating a lead solution by zinc plates. For 1,000 gallons of oil, 30 lb. nitrate of lead are dissolved in 15 gallons of water; about 1 ounce of nitric acid is added, then 6 lb. of sheet zinc. The precipitated lead is placed in capacious funnels plugged with shavings, or, better still, with sea-weed, where it is rapidly washed with water; then a small quantity of oil is poured on very gently, so as to displace the water imbibed by the porous mass of precipitated spongy lead. When the oil runs away clear and limpid from the bottom of the funnel, and as a consequence thereof all the water has been displaced, the mud thus obtained is run into the tank containing the 1,000 gallons of oil, where the whole is subjected to agitation. In working with small quantities the requisite agitation may be imparted by running the oil and lead into a cask, and rolling it about from time to time on the floor. The oil thus treated assumes a reddish tint, which disappears as soon as it oxidises in contact with air. If the treatment has been efficient, the resulting oil is as fluid as the original, contains a small quantity of lead, and dries in twenty-four hours in the open air. The rationale of this process is easily seen, the fatty acids originally present on the oil act on the finally divided metallic lead, and the nascent linoleate of lead, aided by the heat produced by its formation, dissolves in the oil as soon as formed. It is advisable to introduce manganese into the oil prepared in this way. A salt of manganese very soluble in oil, *viz.*, the nitrate, is added,

[1] Liebig's method is regarded by some as simply a method of refining the oil. It was all Liebig claimed for it.—J. G. M.

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say, 15 lb. for the proportions given, and the whole frequently stirred for two or three days, after which it is allowed to stand and decanted. If this oil were at once used, as it often contains an excess of nitrate of manganese, which deliquesces in the air, it might dry dull. To obviate this, 7½ lb. of dry precipitated oxide of lead are added, and the whole again subjected to agitation. After standing a perfectly clear oil is obtained, which, exposed to the open air in a thin layer, at the ordinary temperature dries in six hours. During drying the absorption of oxygen produces, in consequence of the presence of oxide of manganese, a brown coloration, but the latter soon disappears, and finally a beautifully brilliant, perfectly dry and completely colourless coat is obtained. Some manufacturers using this process have found the oil in certain cases to be tacky, perhaps owing to the presence of a small quantity of glycerine. This has led them to heat the oil treated first with finely divided lead and then with a salt of manganese, and it would appear that they have obtained very interesting results. (See pp. 125-127.)

Lewkowitsch, in a recent lecture, has revived the idea that linseed oil can be converted into what is to all intents and purposes a boiled oil by grinding it in the cold with borate of manganese. This would have, in the absence of free fatty acid and free hydrated oxide of manganese, no more effect on linseed oil than grinding it with so much pumice-stone. The writer proved this in an unpublished experiment made some fifteen years ago. He triturated linseed oil with borate of manganese both in the cold and at 100° C. for nearly a week, and the oil scarcely dried any better than raw oil. The spreading of such erroneous doctrine is to be deprecated, notwithstanding that the great Chevreul stands sponsor for it. There is no getting over the fact that the drier must first of all be dissolved in the oil before it can act.

Boiling Oil Through the Bung-Hole.—The American process of making "boiled" oil in the cold, or, as they term it over there, boiling oil through the bung-hole, consists in simply dissolving fused linoleate of lead or manganese, or their rosinates, in spirits of turpentine or naphtha, and adding a certain amount of this solution to each barrel of raw oil. The painter does the same thing when he adds "terebine" to his paint.

Employment of Boiled Oil in Painting.

Boiled oil or oil prepared as just indicated is a very important factor in painting. In fact, in house decoration raw linseed oil is the vehicle used in painting. It is ground along with *white*, that is to say, white lead, so as to obtain a soft paste, which is thinned down in various proportions with linseed oil and spirits of turpentine until the desired fluidity is obtained. If a certain shade is desired, the colouring matter beaten up with a small quantity of oil is added; finally, if very rapid drying is required, what are known as "Patent

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Driers" (siccatives) are added. These may consist of (1) a solid powder (litharge, red lead or zumatic, *i.e.*, a mixture of oxide of zinc and borate of manganese); (2) litharge or manganese-boiled oil when there is no reason to fear the painting, or by a drying oil, the drying properties of which have been stimulated by appropriate treatment (precipitated lead, acetate of lead, etc.); or (3), finally, of special preparations, the base of which is oil known under the name of liquid driers, but which are only used with coloured pigments, as they turn white tints yellow. (See pp. 84-93.)

The paint thus formed slowly changes when spread upon any surface in a thin layer to a solid body, which forms a protective coating against external influences.

The paint ought not to dry too quickly, for the slower it dries the more it is capable of expanding, and thus lasting the longer.

The fluidity ought to be such that the paint flows easily from the brush whilst at the same time it does not run in streams, but forms a homogeneous coating. The coating must also be thin enough to prevent the formation of pellicles at the surface, which would hinder the part underneath from drying. It is therefore advisable to apply several successive coats. But each of these ought to be allowed to dry thoroughly before the succeeding one is applied. With linseed oil substitutes, none of which can possibly form insoluble linoxin on drying, the second coat of paint is sure to disturb the first, especially if applied to outdoor work in warm weather, and if spirits of turpentine or thinners be added to hasten the drying. Substitutes of all kinds should be avoided. They give rise to more worry and annoyance than eventual economy. In some extreme instances one might as well apply methylated spirit to wood coated with brown hard spirit varnish as apply a second coating of these substitutes.

In using boiled oil a brilliant coating is not aimed at, the question of appearance being subordinate to that of resistance and durability. It is preferable to impart brilliancy by a final coat of varnish.

For the grinding of paints in oil, stiff and liquid, see J. Cruickshank Smith's *Manufacture of Paint* (Scott, Greenwood & Co.).

Oil Boiling.

Oil is generally boiled in cast-iron pans, best made in the form of an inverted truncated cone, so that in frothing up or priming the oil may spread over a larger surface. Recently oil-boiling pans have been made of enamelled iron, especially on the continent, but their use is for obvious reasons far from general in this country. In general the pan is half filled with oil and heated for three hours. For some purposes the oil is set on fire a few minutes before the fire is drawn. At other times the oil is heated to a much higher temperature, *viz.*, 200° to 225° C. (392° to 437° F.), and this temperature is maintained, says Livache, in some cases as long as eight days, a seemingly irrational time. So that the heat may not accidentally

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rise too high a small quantity of tin is added to the oil, and care taken that the heat does not reach its melting-point, 228° C. (442·4° F.). Should a viscous or a good bodied oil be required, the temperature is raised to 316° C., say, 600° F., and kept thereat from six to eight hours. But with oils to be used in the preparation of pale varnishes, it is not desirable to heat the oil to too high a temperature, which would impart to it a brown colour and start the destructive distillation of the oil, liberating a certain proportion of linoleic acid, which dries more slowly than linolein. To produce a superior article the heat should not be raised so far as to start destructive distillation or "crack" the oil, which should rather be kept at a temperature which would bring about polymerisation, an object attained by slow boiling at a temperature regulated between 150° and 200° C. (302° to 392° F.). The lower the temperature at which the oil is kept the longer does the process last. According to Livache this is the method adopted in England, where the oil, he says, is boiled for a very long time, often extending over a week; but by working in that way a very pale oil is obtained, presenting great elasticity and possessing properties which more than compensate for the length of time occupied in the process. But as a matter of fact, the bulk of the boiled oil made in Britain is made by some modification of Vincent's process (p. 49), and does not last more than thirty-six to forty-eight hours *at the most*. The point is to get the small amount of manganese into the oil. The air afterwards does the rest.

Treating Oil with Driers.—It has been seen that those which have really a beneficial influence upon the drying of oils are lead and manganese either in the state of metals, oxides or salts. In actual practice the oxides of lead and manganese or their salts are preferred; nevertheless the use of metallic lead has lately been tried with promising results. (See p. 33.)

Driers are generally incorporated with the oil by aid of heat. However, interesting attempts have been made to effect this result in the cold. (See p. 32.)

In 1891 F. H. Thorpe tried to determine the influence of temperature and of the various driers on linseed oil during boiling.

Calcutta raw linseed oil was employed. It had a very pale yellow colour, and had been cold pressed. In each experiment 50 c.c. of oil was used. This was put into a tube of thin Bohemian glass, which was plunged with several others into a sand bath, the sand reaching half-way up the contents of the tube. During boiling the contents of the tubes were stirred. The drier, before being introduced into the oil, was dried. The best results were got by heating the sand bath up to between 230° and 275° C. After heating the quality of the oil was gauged by covering small plates of glass with it and allowing it to dry in a ventilated room. The varnish was considered dry when it could be touched without leaving finger-marks upon it. The following table gives the results obtained. Some of them do not seem altogether in accordance with results obtained on

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the large scale. But results obtained by heating tubes in sand can hardly be regarded as comparative.

Again, the influence of mass in oil boiling makes itself felt as in other chemical operations. Mere laboratory experiments can only serve as a rough guide.

Siccative.	Quantity Employed (in Grammes).	Length of the Boiling (Hours).	Temperature (in Degrees).	Time taken to Dry (Hours).	Appearance of the Dry Coat.
Litharge - - -	1·0	2¼	220	6	Almost colourless.
,, - - -	0·2	2¼	250	10	,, ,,
,, - - -	0·8	1½	250	10	,, ,,
Peroxide of Lead - -	1·072	1½	220	several days	Strongly coloured.
Chloride ,, - -	1·274	2½	250-360	24	Slightly ,,
Red Lead - - -	1·024	2½	220-285	24	Strongly ,,
Oxalate of Lead - -	1·323	2¼	300	did not dry	,, ,,
Tartrate ,, - -	1·6	2¼	270	24	,, ,,
Acetate ,, - -	1·46	2¼	270	12	Slightly ,,
Borate ,, - -	1·105	1½	220-300	20	,, ,,
Carbonate ,, - -	1·197	2	225	10	,, ,,
Oxide of Zinc - -	0·5	2½	250	45	Almost colourless.
Sulphate ,, - -	1·987	2½	285	45	,, ,,
,, ,, - -	1·5	2	230	45	Yellow.
Acetate ,, - -	1·0	2¼	235-280	40	Colourless.
Borate ,, - -	1·0	2	240	40	Almost colourless.
,, ,, - -	0·5	1½	240	46	,, ,,
,, ,, - -	0·5	1½	240	46	,, ,,
Citrate ,, - -	1·5	2½	230	36	,, ,,
Acetate of Manganese -	1·0	2¼	225-250	20	,, ,,
Borate ,, -	1·625	2¼	220	20	Colourless and hard.
Sulphate ,, -	1·72	2	240	40	Colourless.
Oxalate ,, -	1·64	2	230	40	,,
Acetate ,, -	0·5	2	225-250	20	Strongly coloured.
Borate ,, -	0·5	1	230	20	Colourless.
Acetate ,, -	0·5	1½	225-250	20	,,
Oxalate ,, -	1·5	2½	230	36	,,
Sulphate ,, -	1·5	2½	240	36	,,
Oxalate ,, -	1·0	2½	240	48	Yellow.
Citrate ,, -	1·5	1½	230	24	Black.
Tartrate ,, -	1·0	2½	230	24	Colourless.
Formiate ,, -	1·0	1	200	24	Slightly coloured.

The figures in the following table give the amount of metal, whether lead or manganese, in certain well-known driers, the number of pounds per ton of oil to use, and the temperature at which they should be incorporated. The temperature at which the drier dissolves should never be exceeded except with the view of hastening the process in a rational manner (Weger).

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Drier.	Percentage of metal in commercial product.	Lb. per ton of oil.	Temperature. Degrees C.
Manganese Peroxide, MnO_2	30-60 Mn.	—	250
Hydrated Manganic Oxide, $Mn2O_3H_2O$	45-50 „	4½	180-200 better
Hydrated Manganese Peroxide, MnO_2H_2O	45-50 „	4½	200-220
Litharge, PbO	93 Pb.	—	150 generally higher.
Manganese Borate	15-20 Mn.	11-22	In the cold generally
Manganese Borate Commercial	5 Mn.	up to 66	200 and higher.
Precipitated Manganous Rosinate	7	22 to 33	
Fused Manganic Rosinate	4	66	In the cold
Linoleate of Manganese	10	22	generally
Lead Manganese Rosinate	8-10 Pb. 1-2 Mn.	44-66	130-150

Oil Boiling by Naked Fire.—When only small quantities of boiled oil are required, the most simple plan is to boil the oil in an iron or copper pan of about 20 gallons capacity, with an enlarged mouth to prevent the oil from frothing and threatening to prime over. This pan is at a certain height furnished with a collar or circular flange which supports it on the rim of a sheet-iron furnace, fed preferably with wood charcoal. The pot being filled to the extent of half of its capacity with oil, and therefore containing about 10 or 11 gallons, the fire is lighted, and as soon as boiling commences the driers are added in small quantities at a time with constant stirring with an iron rod. The proportion and the nature of the driers used vary much, according to Andés; for the quantity of oil in the pot, either of the following mixtures may be used:—

2¼ lb. of red lead and 2¼ lb. litharge; or
2¼ lb. of litharge and 2¼ lb. sugar of lead; or
1⅛ lb. of red lead and 3⅜ lb. sugar of lead; or
1⅛ lb. to 3⅜ lb. of borate of manganese; or
2¼ lb. of hydrated oxide of manganese.

The driers are previously ground as finely as possible, and the oil well stirred after each addition. As soon as the driers are all in and the frothing has ceased the pot is filled with oil just up to the neck, and the fire regulated so that the temperature does not rise above 220° C. by means of a thermometer with metallic framework. The operation is generally complete in three hours, during which time the driers are frequently stirred up from the bottom. The pot is then withdrawn from the fire, and the oil is set aside to clarify; or if it be desired to start boiling a fresh batch it is run into a galvanised wrought-iron tank.

Though this is a very simple arrangement, it has the great drawback that the pot full of hot oil has to be lifted down from the top of

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the furnace, thus greatly enhancing the risk of accidents. Further, when several consecutive boilings have to be made the heat is badly utilised. To remedy these inconvenient defects a furnace built in a framework of masonry is used. This furnace is sometimes covered with an iron plate, with a circular hole into which the pot fits.

More often, however, the fire is underground, thus enabling the workman to watch the operation at his ease, since the pot is then on the same level as the ground.

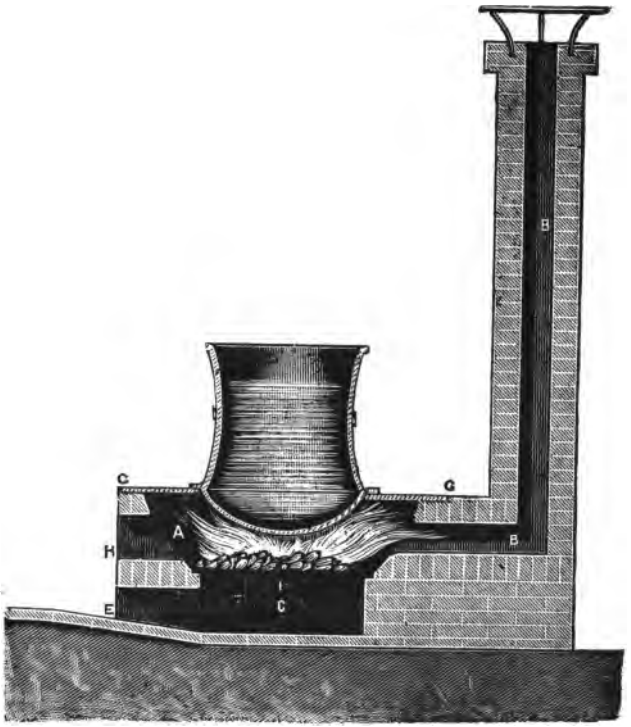

FIG. 11.—Portable Oil-boiling Pot. A, B, flue and chimney-stalk; C, plate on which flange of pot rests.

A rather different kind of pot (Fig. 11) is used, and so constructed that the oil does not receive throughout its whole mass and at the outset the heat stored up in the brickwork. To obviate this the pan of about 20 to 22 gallons capacity, generally of cast-iron, enamelled inside, is contracted in the middle, and rounded at the base, so that the bottom only is exposed to the action of the heat. Otherwise the process is conducted in the same way as before, only the oil is heated a little longer, say, four hours instead of three. This pot is lifted off the fire on to an iron stand (Fig. 12) by two wrought-iron bars, 7 to 10 feet long, passing through loops fixed

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to the side of the pot, so that in case of fire the bearers may be at a safe distance.

When large quantities of oil are to be boiled no good purpose is served by multiplying operations. It is better to use larger pans. In an extensive business the pans may measure 6 feet 6 inches high by 5 feet in diameter. Nevertheless we more often meet with pans having a capacity of 60 to 120 gallons. The pan is of strong iron plate about $\frac{1}{6}$ of an inch thick, is built into masonry in such a manner that the upper part controls a circular gutter of $1\frac{1}{2}$ to 2 inches deep, which, in case of the oil boiling over, conducts it to an adjacent receiver. The pan can be so built into the brickwork that it may be heated on a part of its bottom and its sides, or, on the contrary, and preferably, on its sides alone. In the first case it so rests on the brickwork that the centre part of the bottom is exposed; in the second case, so as not to overheat the bottom part, it is supported by a small arch of brickwork, thus preventing solid matters from tenaciously adhering to the bottom. The *cylindrical* form of pan is not always

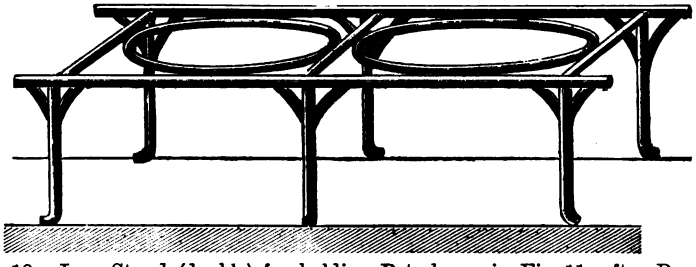

FIG. 12.—Iron Stand (double) for holding Pot shown in Fig. 11, after Removal from Fire.

adopted. We often come across pans almost *conical* in shape, the narrow part of which forms the bottom—a form which has the advantage of distributing the heat more evenly, and, owing to the larger section, of diminishing, in case of frothing, the chances of the oil running over. Such a pan is half or at the most two-thirds filled with oil and at first heated gently, and when the oil reaches 100° C. (212° F.) the impurities on the surface are skimmed off. The driers, previously completely desiccated and ground very finely, are then added. It would not do to add them in the beginning, as they would fall to the bottom, and their effect be to a great extent lost. Andés gives the following proportion for 20 gallons of oil:—

$2\frac{3}{4}$ lb. red lead, $2\frac{1}{8}$ lb. litharge; or
$2\frac{1}{8}$ lb. litharge, $2\frac{1}{8}$ lb. sugar of lead; or
$1\frac{7}{8}$ lb. borate of manganese; or
$2\frac{1}{8}$ lb. hydrate of manganese.

Boiling is kept up for five hours at a temperature not exceeding 220° C. (396° F.). The temperature is regulated by means of a thermometer in metal framework, or, better still, by an aneroid thermo-

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meter, which is more legible, and not so fragile. Sometimes the heat may be regulated by adding some tin, and taking care that the heat does not rise to the melting-point of the latter, *viz.*, 228° C. (442·4° F.). The degree of heat was at one time ascertained by dipping the quill end of a feather, which ought to at once shrivel and curl up, into the oil. This is still a handy test although the ultra scientific may sneer at it. The more attention there is paid to ensure a constant regulated temperature, the brighter and paler will be the oil.

When very quick drying properties are aimed at the oil is boiled from one to two hours longer, and the amount of driers increased

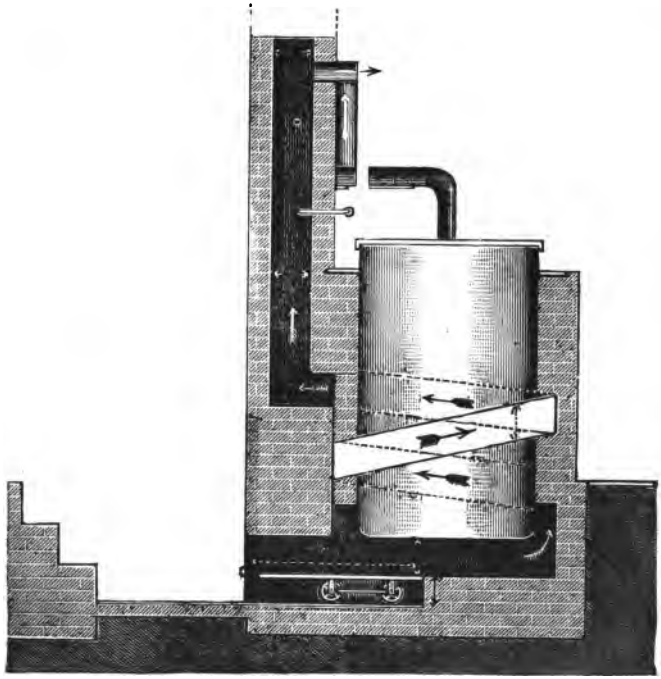

FIG. 13.—Plant for Oil Boiling by Naked Fire (*with Removable Grate Running on Rails*).

from 20 to 25 per cent. At the time of adding the driers the oil should be well stirred either with an iron spatula, or preferably by means of mechanical agitators (a revolving shaft with arms, or a simple hanging chain fixed at both ends to a horizontal shaft, bent twice at right angles, fixed above the pan; the chain descends almost to the bottom of the pan and is turned by means of a crank some distance away).

A wood charcoal fire allows a constant temperature to be maintained if the fire be well regulated. Coke will suit equally well. So as to lessen the risk of the temperature rising too suddenly, which would colour the oil and cause it to overflow or aggravate the risk of

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fire, it has been suggested to use a movable grate, which can be lowered instantaneously, and thus cause the fuel to fall into a pit full of water (see Fig. 15). Movable car furnaces running upon rails (Fig. 14), and thus capable of being easily removed, have also been employed. Oil is sometimes boiled by immersing the pan in a sand bath, or in a bath of a suitable alloy. But this plan is hardly to be recommended, because if the temperature rises too high, the oil cannot be withdrawn from the source of heat; the same thing sometimes happens when the pan is built into masonry or set in brickwork, for even in the case of a movable furnace the oil remains exposed to the action of the heat stored up in the brickwork.

To remedy this the pan, which sometimes measures more than 9 cubic feet, is mounted on a kind of trolley. The circular opening of the fire is level with the ground. If the temperature rises too high the trolley supporting the pan is removed from the fire; a

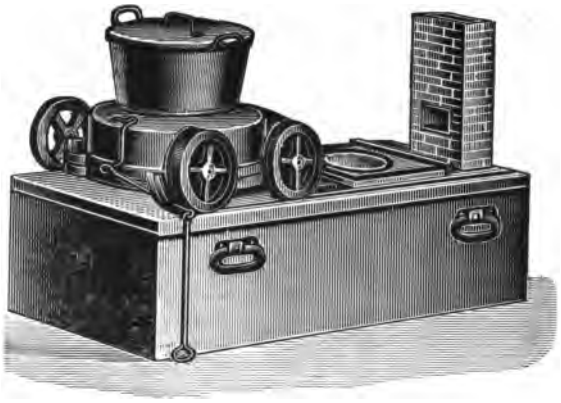

FIG. 14.—Movable Furnace and Oil-boiling Pan.

movable hood with counterpoise descends over the top of the pan and carries away the fumes. This enables the oil-boiler or varnish-maker to watch the operation very easily, and preserves the metal of the pan. When the operation is terminated the oil is ladled out, and on the large scale pumped, into a galvanised iron tank. As soon as the pan is empty it is immediately recharged with another batch, and generally without being cleaned in any way. The coating which forms on the sides of the pan prevents the oil from darkening, but care must be taken to remove any excess of driers from the bottom of the pan, otherwise unintentionally an excess of driers, which might have an injurious effect, may be added to the next batch. When the skin becomes too thick it is scraped off. In certain factories this is only done once a year, or once in two years. The foots from oil boiling by fire may be used in making black paints, which are so difficult to dry.

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Fire Risks and Prevention of Nuisance.—During boiling, pungent, very inflammable, vapours are emitted. Working with small quantities of oil all that is generally deemed necessary to prevent fire is to have a cover at hand to place on the pot should the oil take fire. On the large scale, when the oil is heated in a pan, a hinged lid is fixed over the pan during the boiling by means of a cord. If the oil takes fire the cord burns, and the cover falls automatically on to the pan. Different arrangements have been adopted so as to mitigate the fumes. On the small scale, a simple hood connecting with a chimney-stalk of sufficient elevation and draught is placed above the pot. A better plan is to fix a sheet-iron cap above the pot (Fig. 15), provided with

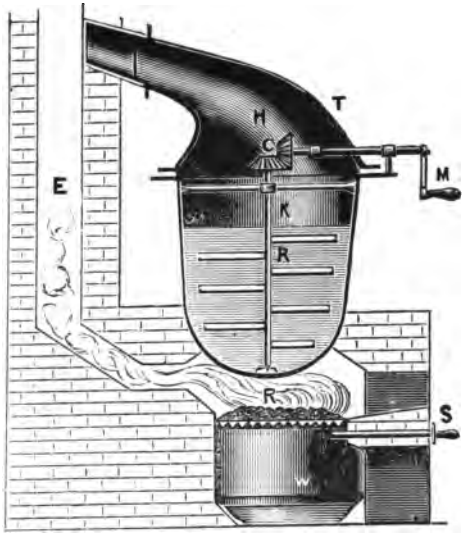

FIG. 15.—K, Oil-boiling pan, with fire-quenching arrangement; R, agitator with blades; M, crank-handle working cog-wheel gearing, C; H, hood to convey fumes to chimney-stalk, E; R, hearth, the bars of which are kept in position by the rod S, by means of which, if the oil threatens to prime over or catch fire, the furnace may be extinguished by tilting its contents into the vessel W, filled with water.

an opening in front by which the process may be watched, or the contents of the pot stirred. This cap is connected by means of a pipe with a tall chimney shaft, the lower part of which is closed by a plug. Here thick viscous products with a vile odour condense. They are removed from time to time by opening the plug. The more volatile products escape from the top of the chimney. But it is better not to let the fumes escape directly into the air, but to pass them through a condenser in connection with the chimney. A very simple way is to cover the boiling pot with a movable lid, with a window in the centre capable of being opened so as to allow of stirring the oil. On the side is a disengagement tube, which can be

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luted to a pipe connected with a condenser. The latter may be a pipe, shaped like an organ pipe, leading into a vessel containing water, or it may consist of a vertical cylinder, the interior of which contains a coil of piping, through which cold water circulates. The vapour passes through the space *not* occupied by the pipes, and is in a great measure condensed. The advantage of this is that the wide, straight cylinder is not so liable to get choked up. It is advisable to use an aspirator because the vapours are very heavy, but working with one or other of these arrangements condensation is almost complete. If need be the uncondensable vapours may be led through a fire to burn them, as shown in Fig. 16 (p. 47). Often no previous condensation is attempted, and the fumes are led directly into the fire. At first it was supposed that explosions would occur, but the large quantity of air which is mixed with the fumes does away with any danger. It is, however, necessary to be very careful that the oil in the pan does not catch fire when these products are conveyed directly to the fire which heats the pan, or to a special furnace. The oil may take fire by the heavy vapours condensing in the pipe almost as soon as they leave the pan, and should there be any flaw they may run slowly backwards towards the fire and there become inflamed, and thus heat to redness the connecting pipe, which is generally of sheet iron, and thereby set fire to the condensed products in proximity to the boiling oil, and thus to the oil itself. This is easily remedied by protecting the lower part of the conduit pipe by fire-clay bricks, and adding a damper, so that communication may be shut off should the temperature rise too high. If the fumes be condensed by means of a properly constructed condenser between the pan and the fire, no fear need be entertained.

Choice of Drier—Manganese Driers.—It has been shown that the best results are obtained by the use of lead and manganese. In particular, an oil boiled in contact with manganese dries quicker than one boiled with lead. When boiled oil is free from lead it has the advantage of not being altered by sulphur compounds. The receipts given for the different driers to be added recommend the use of the borate and the hydrated oxide of manganese to the exclusion of all lead compounds, and the boiling may be done as suggested. The superiority of oils having manganese as their only drier is so decisive that attempts have been made to manufacture such oils directly.

1. *Borate of Manganese.*—Two pounds of very white borate of manganese, free from iron and finely ground, are added with constant stirring to 1 gallon of oil heated to 100° C. (212° F.). When complete incorporation is effected the heat is raised to 200° C. (392° F.). One hundred gallons of oil are heated in a pan until bubbles of gas commence to come off, when the preceding mixture is added in a thin stream. The heat is raised to about 200° to 220° C. (392° to 428° F.), and after twenty minutes' boiling an oil is obtained which rapidly dries to a bright elastic coat. It is absolutely necessary only to use

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borate of manganese completely free from iron. It is prepared, according to Livache, by adding a solution of borax to a solution of chloride of manganese, so long as a precipitate is formed which is washed with water and dried. A perceptibly white product is thus obtained.

As manganese ores are liable to contain iron, lime and other impurities, this method of preparing borate of manganese is apt to give an impure product; a better method is that given on pp. 89-90. The drying properties of this salt are such that it is claimed by some that it can transform linseed oil into a quick-drying oil at a temperature as low as 40° C. (104° F.), *e.g.*, if in a flask containing linseed oil we suspend a small linen bag containing borate of manganese (3 oz. of borate to 100 oz. of oil), and if we place the flask in a warm place we obtain in fifteen days a quick-drying oil. But all these trivial processes are misleading. Life is too short to wait fifteen days for oil to dry no quicker than boiled oil, and only under exceptional conditions, such as the presence of free fatty acid in the oil and free hydrated oxide of manganese in the borate, can the oil be acted on at such low temperatures. Common-sense might tell those who make such extravagant claims for borate of manganese that in the cold it is as inert as ground pumice-stone or sand. (See also p. 34.)

Yet we obtain a better oil if, instead of employing borate of manganese alone, we employ a mixture of borate of manganese and oxide of lead, which is practically the same, because the reaction which ensues introduces oxide of manganese, but the latter may be added directly in the form of the hydrated oxide—a dense, brown product; finally it can be introduced by utilising reactions similar to the following:—

2. *Hydrated Oxide of Manganese and Air.*—One thousand pounds of oil are heated to 70° to 80° C. (158° to 176° F.); on the other hand, 3 lb. of crystallised sulphate of manganese are dissolved in a very small quantity of water by the aid of heat; 10 lb. of caustic potash dissolved in a small quantity of water are added; the whole well stirred and run into the oil. The mass turns brown, and brightens at the same time. An india-rubber tube, to the end of which a rose nozzle is fixed, is introduced into the pan, and by means of a pump air is injected for four or five hours until the brown coloration has disappeared.

It has likewise been proposed to cause the oil to which the above mixture has been added to fall in drops inside a pipe, where a current of air circulates the reverse way. The oil is again elevated, and made to fall again, and so on until the brown coloration disappears. (Fig. 22, p. 58 and context.) It has been further proposed to use the natural black oxide of manganese—pyrolusite.

3. *Black Oxide of Manganese and Sulphuric Acid.*—A mixture of 2 lb. of finely ground pyrolusite and 2½ lb. of sulphuric acid is added to 100 lb. of linseed oil heated between 180° and 200° C. (356° to 392° F.). After about an hour, a milk of lime, obtained by slaking

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1 lb. of quicklime, is added, the whole well stirred, and filtered through canvas.

4. *Various Processes.*—(*a*) Livache claims that a very quick-drying oil—free from lead—whose drier is manganese may be obtained in a roundabout way by slightly heating or simply agitating an oil which has been boiled with litharge, with borate or sulphate of manganese. The oxide of manganese, he says, replaces the oxide of lead, which is precipitated in the state of insoluble borate or sulphate.

(*b*) One hundred gallons of linseed oil, for example, are heated with 50 lb. of oleate of lead. It has been proposed to replace the oleate of lead by the linoleate, or more recently by the rosinate of manganese, which are used in smaller proportion.

Zinc Driers.—The oil is sometimes boiled after the addition of white lead, acetate of lead and sulphate of zinc. The function of zinc sulphate in oil-boiling and varnish-making is obscure; it is generally added with the idea of imparting elasticity. Again, attempts have been made to substitute lead salts by pure oxide of zinc, but, after boiling, the resultant oil dries slowly, and has no advantage over a litharge-boiled oil except that it is not blackened by sulphuretted hydrogen. It is much more profitable, says Livache, to precipitate the lead of a litharge-boiled oil by means of a suitable manganese salt such as the sulphate, but this is a very roundabout way of introducing manganese into the oil.

In certain cases it has been recommended to add oxide of zinc to the salts of manganese employed in oil boiling, but if the oxide of zinc did any good in these cases it was due to the liberation of oxide of manganese. Leaving out of account the advantage—often very appreciable—of obtaining an oil which does not blacken in contact with sulphuretted hydrogen, it would appear that the oil does not dry quite so quickly, nor so uniformly and well, as when a salt of manganese is added in presence of oxide of lead.

It has been recommended not to heat the oil with driers beyond 120° to 127° C. (248° to 260·6° F.), but to project into the oil after several hours' boiling $\frac{1}{400}$th of its weight of water, mixed with $\frac{1}{50}$th of its weight of litharge, in the form of fine drops. But this process, due to Bartky and tried in Germany, appears to be but little used.

Oil Boiling by Steam.—There are two great drawbacks to boiling oil over a naked fire, *viz.*, the danger of fire and the dark tint which the oil assumes in contact with overheated surfaces. It has been tried to remedy these by boiling oil by means of a steam heat, and this method of oil boiling ensures perfect safety, economy and rapidity, advantages which are the more to be appreciated because this method produces pale oils. The simplest plan is to heat the oil in a pan by means of a *steam coil.* Steam at about 4 to 5 atmospheres (60 to 75 lb. pressure) is passed through the coil so as to heat the oil to about 150° C. (302° F.), but a much lower temperature is sufficient, say, about 40 lb. steam. A mechanical agitator spreads the heat uniformly through the bulk of the oil. Working with a

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pan capable of holding half a ton of oil, when the oil has been heated for half an hour the necessary quantities of driers are added, and the boiling continued for six hours, the mechanical agitator being kept at work all the time. The process is, says Livache, rendered more economical by using superheated steam. In this case the steam before entering the pan passes through a coil in a furnace capable of bringing it to 400° C. (752° F.). When the superheated steam in its passage through the coil in the pan has brought the oil to the desired temperature, it is then only necessary to maintain it at that temperature by means of the steam admission valve. But there is no necessity for this superheating, 40 lb. steam does all that is required. It is not intended to distil the oil, and unless this were so there is no reason for superheating.

To work as economically as possible, the gases evolved from the furnace in which the steam was superheated are led underneath the

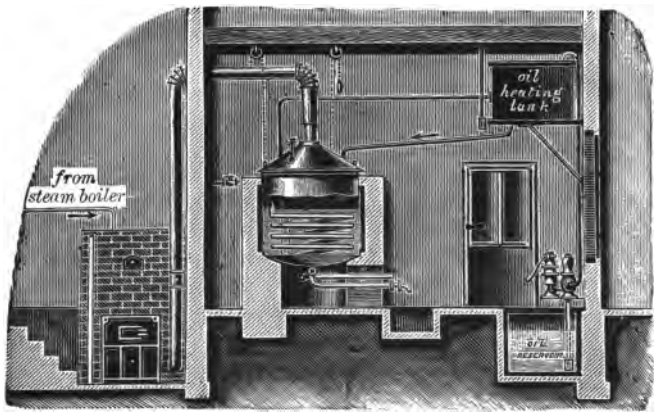

FIG. 16.—Fireproof Oil Boiling by Superheated Steam and Fume Consuming Plant.

pan and burnt so as to assist in heating the oil. But if this arrangement be economical, it introduces the element of danger from fire, the obviating of which we had, amongst other advantages, in view when adopting the process of oil boiling by steam. In boiling oil by a steam coil there is always the serious drawback that there is a great difficulty in maintaining a uniform temperature throughout the mass even with continuous agitation. Effectual commingling of the oil and driers is not practicable; the latter settle to the bottom, and the coil of piping prevents the agitator from reaching them. Were it not for this drawback linseed oil could be very well boiled in a comparatively shallow, lead-lined, rectangular wooden tank lined with 6 or 7 lb. lead. This drawback, however, could be easily got over by pumping in air through a perforated pipe laid down so that each coil thereof alternated with a coil of the closed steam pipe. This, in conjunction with the mechanical agitator, would effectually prevent the

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drier from settling at the bottom. By suitable mechanical arrangements a boiling pan made in this way would be equally effectual and cost less, not only originally but also for maintenance, than the costly wrought-iron or copper vessel now in vogue. But certain manufacturers deprecate the continuous stirring of the oil as likely to produce bad results.

Andés obtained good results by using a steam-jacketed pan (Fig. 17), 85 gallons capacity, made from strong iron plate, $\frac{1}{5}$ inch in thickness, and able to stand a pressure of 4 or 5 atmospheres; it is provided with a safety valve, blow-off cock, and a tap to run off condensed water. About 40 gallons of oil are run into the pan, the steam turned on, and when the pressure is sufficient the blow-off cock

FIG. 17.—Steam-Jacketed Pan for Oil Boiling.

is opened for a minute to allow the steam and air to escape; this is repeated several times, and the escape valve adjusted, so that only the small quantity of spent steam, the replacement of which by superheated steam is necessary to keep the pan and contents at the desired temperature, is allowed to escape. The condensed water is run off when necessary. The steam is regulated so as to keep the temperature between 125° and 132° C. (257° and 269·6° F.), and the process lasts from five to six hours, but by prolonging the boiling and using more driers better quality oil is obtained. If care be taken not to exceed 132° C. (269·6° F.) the resultant oil is very pale and bright. To stimulate oxidation during the boiling process, Andés fixes a mechanical agitator (R, Fig. 19) on the surface of the oil, which is propelled very

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slowly. The same driers may be used as in boiling oil by fire, but it is advantageous to alter the proportions. Only half of the quantities of lead compounds should be used, but double the quantities when working exclusively with hydrated oxide of manganese or manganese salts. To economise, two steam pans may be wrought together, the steam passing from the steam-jacket of the one to that of the other (Fig. 19, I. and II.). But it is perhaps more advisable to utilise the waste steam (as shown in Fig. 16) so as to heat in an intermediate tank the next batch of oil as it comes from the settling tanks, and

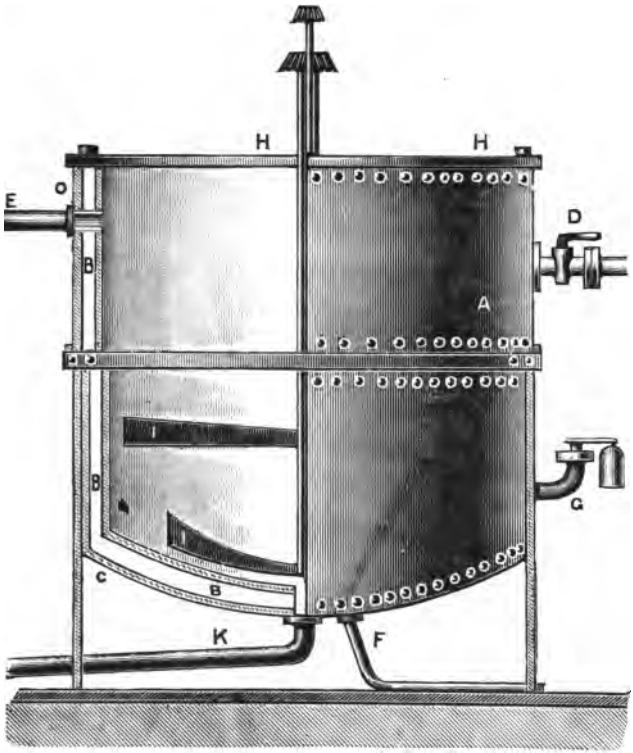

FIG. 18.—Steam-Jacketed Pan with Agitator for Oil Boiling, etc.

previous to the actual boiling. This preliminary warming almost does away with the frothing of East Indian linseed oil, and the consequent liability of the oil to prime over into the condenser when it is heated in the jacketed pan in the usual way.

Vincent's Process for Oil Boiling by Steam with Simultaneous Injection of Air.—In Vincent's process a pan, preferably of copper, is used—having a depth equal to its diameter, of 2 to 10 tons capacity, and surrounded with a steam-jacket, E, up to three-quarters of its depth, or to the oil level or a little above it, and capable of withstanding a working pressure of 40 lb. to the square inch—into the interior of

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which the steam is led, as a source of heat, and so regulated as to register throughout the process a pressure within the jacket of 35 lb. to the square inch. The top of the pan is closed by a dome riveted to it, and pierced with a manhole. In the centre is a stuffing-box through which pass two concentric vertical shafts—a hollow shaft encircling a solid one—armed with blades. By means of exterior gearing (Fig. 20, B) these fans turn in opposite directions, and in so doing intersect each other, and thus by their "dashing and cutting" action cause energetic agitation of the oil and thorough incorporation therewith of the driers. From the dome the fumes are either led by a 6-inch pipe to a condenser or into the furnace for heating the steam boiler. Finally, as soon as the pressure of steam

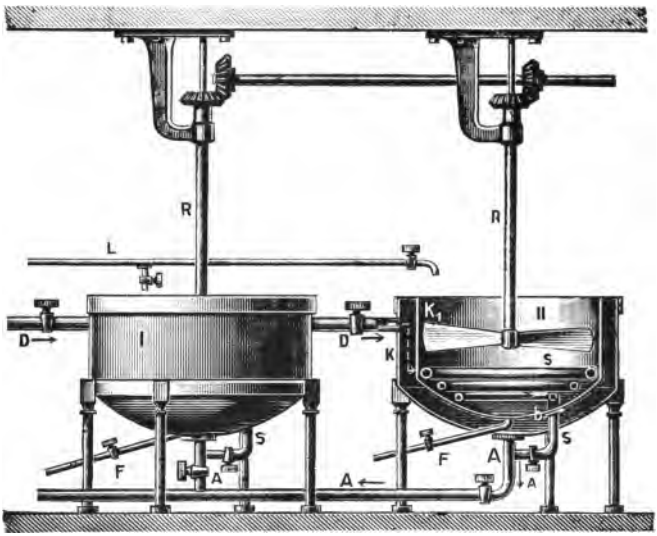

FIG. 19.—Steam Oil-boiling Pans, showing how to connect up series from same steam supply pipes; I., exterior view; II., section; R, agitator; KK_1, steam jacket; S, steam coil; L, raw oil charging pipe; F F, boiled oil discharging pipes; D D, steam inlet pipes; S A, condensed water and waste steam exits.

on the pan registers 35 lb. to the square inch, equal to a temperature of 126·8° C. (260° F.), air is injected under pressure by means of a pipe passing through the jacket into the bottom of the pan.

The oil to be boiled at one operation is first pumped into a large reservoir (Fig. 16), where it is stored as long as possible; from this reservoir it is run into an intermediate tank (Fig. 16) traversed by a 1½-inch coil of iron piping through which the waste steam from the pan passes, thereby economising expense besides facilitating the deposition of impurities. The oil thus heated to about 35° C. (95° F.) is pumped or run by gravitation (see Fig. 16) into the pan, and steam turned on in the jacket. As soon as the pressure marks 2 atmospheres, 35 lb. to the square inch, the agitators (Fig. 20) are put in

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motion and air pumped in. The finely pulverised drier, beaten up with a little oil to the consistency of treacle, is introduced into the pan slowly and in a thin stream through a funnel with a stop-cock fixed in the dome as soon as the oil in the pan has become evenly and thoroughly heated through its bulk, or about half an hour after the steam indicates a pressure of 35 lb. Introducing the drier in this highly comminuted condition prevents coagulation, and thus gives practical effect by complete diffusion throughout the oil to Vincent's theory of oil boiling by steam, which requires each particle of oil to be in contact with, or in proximity to, a particle of the drier used and the oxygen of the air at the same time. Air is injected in this way

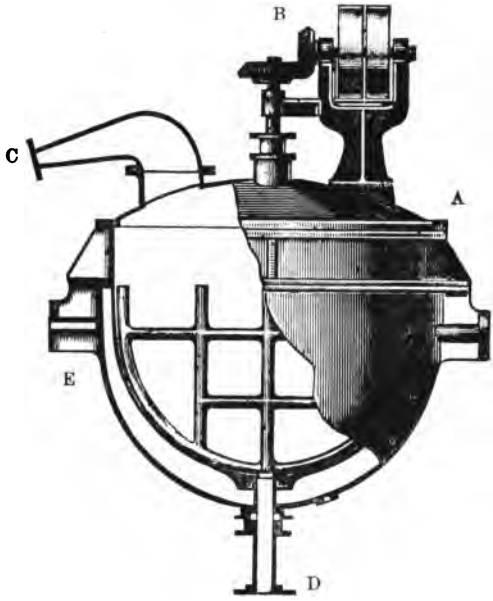

FIG. 20.—Jacketed Pan for Oil Boiling, with dome, A, fitted with agitator driven by cog-wheel gearing, B, and pipe, C, leading to condenser.

so long as it is absorbed and does not cause the oil to froth up and prime over into the condenser. It was thought at first it might be advisable to heat the air before injecting it into the pan, but this would appear to be useless, as it becomes so heated by the pressure to which it is subjected in overcoming the weight of the oil, lifting the heavy valves, etc., that the pipes through which it passes cannot be held in a firm grip in the hand, and not only so, but its passage through the oil cools the latter but very little. The process lasts four hours. By means of a 2-inch pipe (D, Fig. 20) in the bottom of the pan the oil is run into tanks (Fig. 21) where it deposits any undissolved driers, etc. It is through this discharge pipe that the air is injected into the oil. Injecting it in any other way leads to the

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injection pipe getting choked up with oil-skins, etc. Boiling oil by Vincent's process is especially applicable for use with the rosinates and linoleates of manganese. Vincent left us no record of the drier he used, possibly it was the borate of manganese; but the solution given under manganese acetate (p. 89) is an excellent drier for use in this process in the proportions stated.

Boiling by Superheated Air.—Notwithstanding the advantages incidental to the process of oil boiling by steam, there is one drawback which has been considered so serious by some manufacturers as to lead them to give up this method. They found that even with superheated and thoroughly dried steam there was always a certain amount of moisture which acted on the metal of the pan, piercing it in holes and causing bursting or explosions. Superheated air was therefore substituted for superheated steam. This air is drawn by means of a fan from the superheater to the (steam) coil or jacket, and is again returned to the superheater, thus working in a continuous cycle. The difference between the specific heat of steam and that of air renders the economy of the hot-air process very problematical. The coil in the pan is made of copper, which, although dearer than an iron one, has the advantage of not colouring the oil. As copper is also attacked by hot oil, it has been recommended to electro-plate it with silver. The coating need only be but very thin, as it undergoes no wear and tear.

Rules for, and Changes which take Place in, Oil Boiling.—Three points require attention: (1) The amount and nature of the driers; (2) the temperature of boiling, and (3) its duration. When working with naked fire, the quantity of drier varies from 2 to 4 per cent. of lead compounds and 1 to 2 per cent. of hydrated oxide of manganese. The deposit which forms at the bottom of the pan consists of a viscous mud, and when a drier with a lead base has been used, we also find a notable quantity of *metallic* lead, whilst the boiled oil itself only contains about 1 per cent. of *oxide* of lead. It follows: (1) That the oxidisable principles of the oil have reduced a certain quantity of oxide of lead; (2) that a certain quantity of oxide of lead is thrown down in the form of lead salts insoluble in oil; (3) that a part of the oxide of lead enters into a soluble combination with the oil; this combination takes place by the simple saponification of certain glycerides of the oil, and consequently a corresponding quantity of glycerine is liberated. These different reactions are set up from the commencement at a moderate temperature. In the case where manganese is employed instead of lead, the boiled oil contains about $\frac{1}{2}$ per cent. of oxide of manganese.

The temperature ought to be regulated between 210° and 228° C. (410° to 442·4° F.) so as to get a good boiled oil, because (1) oil when heated commences to give off volatile products at about 230° to 236° C. (446° to 456·8° F.), with the formation of fatty acids which yield oxidation products of inferior quality, requiring longer time to dry. When it is required to produce a limpid oil with but little body

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(which is not always the case; take printers' ink, for example), it is advisable not to go beyond 230° C. (446° F.). (2) Again, as a consequence of the saponification of a small quantity of oil a corresponding quantity of glycerine is liberated; now if this glycerine remained, in the free state, in the oil the oxidation products would never dry perfectly but remain tacky. The glycerine must therefore be eliminated; this result is obtained by prolonged boiling, for the glycerine is partially carried away by the volatilisation of certain principles of the oil, which are given off in small proportion during the whole process, however carefully regulated, and even although the temperature remains lower than the temperature of distillation of glycerine. Another portion of the glycerine decomposes under the action of prolonged heat, either in presence of metallic lead, as found at the end of the operation, or into volatile products such as acrolein, acrylic, formic, or acetic acids, and finally the larger quantity reacts upon the triglycerides of the oil, forming diglycerides which afterwards oxidise and dry perfectly. Now this latter reaction in which one molecule of free glycerine combines with one molecule of a triglyceride to form two molecules of a diglyceride, says Livache, occurs at 200° C. (392° F.). As a consequence of these facts, he says, it follows that to get good results the temperature should not be allowed to get below 200° C. (392° F.) nor rise to 230° C. (446° F.). In actual practice a temperature of 220° and 228° C. (428° to 442·4° F.) is aimed at.

Finally, the process lasts from three to six hours according to the size and shape of the pan, and the manner in which the furnace is built on which the pan is heated. The oil ought to be boiled long enough for the above reactions to take place, but the boiled oil obtained as a final result will be better in quality the longer it has been boiled, for on prolonged boiling a polymerisation of the glycerides takes place, giving a final product which oxidises more rapidly and is very elastic after drying. According to Livache, this is the reason why in England, instead of boiling rapidly so as to "break" the oil as in France, the operation is carried on very slowly at the lowest possible temperature, the boiling proceeding for several days, sometimes for even a week. But, as already mentioned, the fire boiling of oil is long out of date in Britain.

When the oil is boiled by superheated steam, or superheated air, the temperature is regulated between 125° and 130° C. (257° to 266° F.). When working with steam, the latter is superheated to 4 or 5 atmospheres, relaxing in the steam coil or jacket to about 2 atmospheres. It will be readily understood, looking at the matter from the double point of view of safety and economy, how difficult it would be to superheat the steam to such a temperature that the steam in the coil or the jacket would be at 200° C. (392° F.), because it would be necessary to heat the oil to 15 atmospheres. Consequently the liberated glycerine remains to a larger extent in the oil, and thus prevents it from drying so well as fire-boiled oil. However, as the

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oil boiled in this way has the advantage of being very pale, endeavours have been made to get over the difficulty, either by using a larger proportion of manganese drier, so as to have a more energetic oxidation and thereby a splitting up of the glycerine, or, preferably, by producing direct oxidation by a current of hot air, or finally, by causing a current of air to circulate through the whole mass of heated oil; or by projecting on the surface of the oil a small quantity of water so as to carry away the glycerine in its train. However this may be, it is most likely that we have here the reason why many persons in the trade prefer oil boiled by fire-heat. Nevertheless, prolonged boiling by steam diminishes and even does away altogether with this disadvantage.

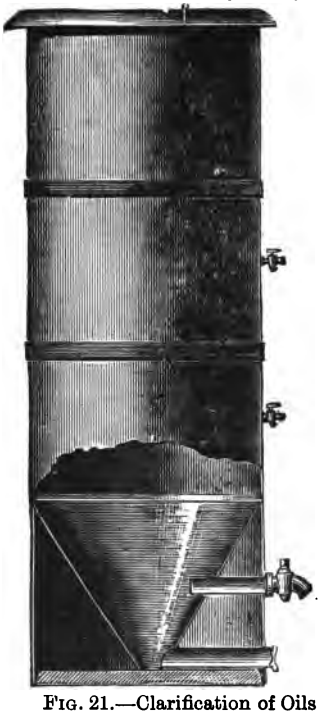

FIG. 21.—Clarification of Oils by Deposition. Tank for storing oil, showing conical arrangement for deposition of foots and draw-off cocks at different heights. (Donovan & Co., Ltd.)

Clarification of Boiled Oil.—When the boiling is finished the oil contains suspended solid matter—coagulated or carbonised organic matter, insoluble salts, undecomposed or unreduced drier, etc. In order to hasten deposition the oil ought to be left to rest as hot as possible, because it is then more fluid and limpid, thus facilitating the more rapid deposition of extraneous matter. It should therefore be run from the boiling pan into the settling tanks as soon as the steam is turned off the pan. When working with small pans which can be easily handled, all that has to be done is to lift them off the fire and to let them stand for eight days at least. On the other hand, when working with large-sized pans built in masonry, they must be emptied quickly, either by a ladle or by a pump, as hot as possible, into a reservoir of the same size, where it is left to deposit for a fortnight at least. Here it may be remarked that there need be no difficulty in drawing hot oil from one vessel at a certain level to another vessel at a lower level by means of a lead-pipe syphon, which should be perfectly dry and filled with *warm* oil and held in the hands by a piece of stout felt if it be desired to touch it after it is set. In setting it the charged syphon should be brought as close to the surface of the hot oil as possible before letting go, or the end of the syphon may be plugged up and the plug driven out after it has entered the oil. After standing for a fortnight the decanted oil may be sent into the market. If stored too long before being used, the oil, at first limpid, becomes cloudy, or at least dull, when knocked

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about in moving or transported in barrels. This is due to chemical changes, which supervene slowly throughout the mass. All that has to be done is to allow the oil to stand for a day or two after it has reached its destination, and to decant cautiously. The amount of deposit formed in this way should not exceed 1 to 2 per cent. of the oil.

Some oil boilers, who ought to have known better, have been known to fill the boiled oil into drums for despatch abroad after scarcely twenty-four hours' settling, and that after boiling with 10 per cent. of cotton-seed oil! It need scarcely be remarked that goods made in this way are liable to be returned, and orders lost never to be regained.

When cotton-seed oil is cheaper than linseed oil it is sometimes attempted to boil it in the proportion of 10 to 20 per cent. with linseed oil. An oil produced in this way will never clarify, leaving altogether out of account its diminished drying properties. Its very questionable recommendation is that it is an excellent way of producing a superabundance of boiled-oil foots. Cotton-seed oil is, in fact, only classified as a drying oil to fit in with the ideas of theoretical chemists, who would fain make practice and actual facts subservient to theory. They choose to ignore the fact that stearine abounds in cotton-seed oil, and that nothing will prevent the stearate of lead and manganese from continuously settling out *ad infinitum*.

The very prolonged storing of *boiled* oil, far from improving it, deteriorates it, especially in the case of lead-boiled oil. After from five to ten years the oil separates into two fractions, the one solid and brittle, whilst the other, retaining its fluidity, can still be used. Oils containing manganese alone do not alter in this way. The deposit formed during oil boiling varies, according to the quality of the oil and the boiling process adopted, from 5 to 8 per cent. of the quantity of oil treated. It is of medium consistency, and, according to the quality of the oil, the colour varies from white to yellow. Andés states that impure, bad quality oil gives a white or yellow deposit, whilst well-purified good oil yields a brown one, with no granular or crystalline appearance. The *loss in oil boiling* and the cost may be compensated by adding 2 or 3 per cent. of thick rosin oil to the raw oil before boiling. This percentage does not affect the finished oil to any great extent. If the boiling process has been well conducted, *i.e.*, at a temperature not exceeding 220° C., this deposit constitutes the only loss. It is carefully collected, being worth sometimes as much as 50 to 60 per cent. of the oil used. It finds a use in the manufacture of mastic (cements), soap and low-quality paints, especially low-quality blacks, the tint of which it does not injure, whilst it greatly improves their naturally bad drying properties.

Properties of Boiled Linseed Oil.

Colour and Fluidity.—If we take linseed oil as the type of a drying oil, it ought after boiling to be a little less fluid than the raw oil, and vary in colour from pale yellow to deep yellow, or,

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at the furthest, reddish brown. *Smell.*—The smell, without being pleasant, should not be nauseous. *Taste.*—Taste similar to that of the raw oil, bringing to mind the vapours given off during boiling. A pungent bitter after taste on the back part of the palate indicates rosin oil. *Density.*—According to Livache, an oil marking 29° with the Fisher and Bex's oleometer, equal to a density of ·9325, will register, on the other hand, after being well boiled with lead, 24°, equal to a density of ·9433, or after boiling with manganese, 26°, equal to a density of ·9389. But the density of a very good boiled oil made on the large scale in England according to Vincent's process, and drying to a non-tacky, firm, elastic, durable coating in six or seven hours in fine weather, and containing 10 per cent. of rosin oil, but no other impurity, has always been found to have a density not exceeding ·934, and if the process be well conducted the oil in question will dry in six or seven hours in summer, and about ten in winter. Those writers who get in raptures over a boiled oil drying in twenty-four to forty-eight hours simply make themselves ridiculous.

Driers : Detection of.—To detect in a boiled oil the substances used as driers, Livache gives the following methods : A small quantity of the oil is poured into a test tube and shaken with dilute sulphuric acid. A white precipitate becoming black on the addition of sulphuretted hydrogen indicates lead. If no precipitation takes place, and if the sulphuric acid becomes green, blackening by the addition of sulphuretted hydrogen, the oil contains copper compounds. When sulphuric acid gives no precipitate, if the acid be neutralised by ammonia on adding ammonium sulphide, iron gives a black, manganese a flesh, and zinc a white-coloured precipitate ; further, in the case of zinc, the acid solution gives a white precipitate with ammonia, soluble in excess. If, instead of using ammonia, we neutralise with carbonate of soda, iron yields a green precipitate, blackening in the air, and the original solution becomes blue on the addition of yellow prussiate of potash ; manganese gives a white precipitate blackening in the air.

Ignition Method.—The better plan is, however, to burn the oil with the usual precautions observed in the ash determination of vegetable substances, and after weighing the residue on ignition to make a systematic qualitative and then a quantitative examination thereof. Zinc oxides and salts are, however, liable to volatilise and escape recognition. The methods given for the determination of mineral substances in oils in books devoted to the chemical analysis of oils are quite unreliable, and none more so than those of Benedikt. A *systematic* examination of the ash is the only reliable method. It is most unscientific to estimate lime in the analysis of the ash of oil in the same way as is done in the case of marble ; we know beforehand that the marble is almost chemically pure carbonate of lime. As to the ash of an oil we know nothing whatever, and to proceed to its analysis with preconceived notions shows very bad training on the part of the analyst, whoever he may be.

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CHAPTER III.

LINOLEUM MANUFACTURE.

Thickening of Linseed Oil by Oxidation.—Whatever process we may adopt in the boiling of oil, the consistency or body of the resultant product is but little different from that of raw oil. Now, in many cases it is very advantageous to use an oil of good body, so as either to hasten the drying when it is used either directly or after it has been incorporated with varnish, or when it is desired to apply the oil to porous surfaces, wood, etc., without having reason to fear of its being imbibed by such substances. The simplest process consists in causing the oil to fall in a thin layer, in contact with air, as we have seen in the cold process of making drying oils (p. 32), but the process is a long one, and oxidation becomes difficult as soon as the oil attains a certain thickness, even when working at a temperature of 50° to 60° C. (122° to 140° F.), by which the oil is kept fluid for a longer time. Further, a quantity of oil adheres to the plates, and there solidifies, causing a serious loss when it is not desired to convert the whole into a solid body. Finally, after a time a solid pellicle is formed on the surface of the oil, which prevents oxidation, and consequently the thickening from proceeding through the mass of oil.

Attempts have been made to increase the fluidity of the oil artificially by dilution with a suitable solvent. By taking, *e.g.*, equal parts of manganese-boiled oil and benzine, and stirring the mixture in a closed vessel, with simultaneous injection of air, rapid absorption of oxygen takes place. This absorption is stimulated by aid of heat regulated between 40° and 50° C. (104° to 122° F.). If the air be continuously injected so as to supply the quantity of oxygen necessary for complete oxidation of the oil, a quantity which amounts at least to 16 per cent. by weight of the oil used, the mixture soon thickens, and if finally we distil off the solvent a viscous residue is left on cooling. If the process be carried on so as to completely oxidise the oil, the liquid becomes cloudy owing to the formation of linoxin, which is insoluble in benzine; on separating the latter, the linoxin is obtained as a very dry, elastic solid. In actual practice boiling is continued for a long time, often for three or four days, and the temperature pushed as far as 250° to 300° C. (482° to 572° F.). By this means heavy bodied oils are produced, which can be thinned down by the

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addition of thinner bodied oil, boiled only from six to eight hours at 200° C. (392° F.). But boiling at so high a temperature has the disadvantage of liberating fatty acids, which take longer to oxidise, and render the oil fatty. In England and Germany preference is given to boiling oil by superheated steam or superheated air either in pans as flat as possible, so that the surrounding air may have free access, or in deep, closed pans, into which either cold or hot air is injected. In certain manufactories, especially in Holland, no drier is used; it is considered preferable to oxidise the oil exclusively by the injection of hot or cold air. The product is superior, but the process rather slow. S. Lewiak has taken out a patent for the injection of hot air in the production of linoleum, No. 14518, 1903.

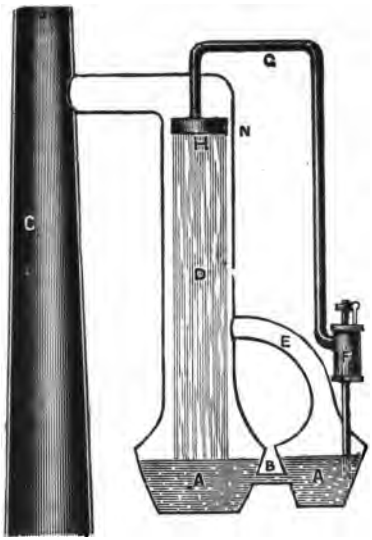

FIG. 22.—Linseed Oil Oxidation Plant. AA, vessels containing oil communicating through B; H, reservoir with perforated bottom; N, pipe with upward current of air; D, downward shower of oil; C, chimney-stalk; E, air pressure equaliser; F, pump.

Storage of High-class Boiled Oil.—According to Livache, when a choice boiled or thickened oil is manufactured, it should not be stored either in iron or copper vessels without electro-plating the interior with silver; this deposit need not be thick, as it is subject to no wear and tear, and owing to the low price of silver would not be so costly as would at first sight appear. The same result may be arrived at, *viz.*, prevention of discoloration by a layer of enamel which stands well and is economical. Wrought-iron vessels "galvanised" on the inside surface with tea lead which contains tin have no action on boiled oil, varnish, nor on spirits of turpentine. One per cent. of tin added to the lead has the same effect.

Walton's Processes.—In F. Walton's, Lion's, Hadfield's and other processes, air is made to act upon oil falling in drops, in such a manner as to present a large surface to oxidation. The *modus operandi* of such processes will be understood from Fig. 22. Walton's plant is composed of a reservoir, the bottom of which, pierced with a number of holes, rests upon a second rectangular reservoir, of which two of the faces are made of glass so that the action of sunlight may intervene with the view of stimulating oxidation and bleaching the oil; the other two faces are made of perforated sheet zinc; through one of these faces air enters, the whole way up the column, and passes out through the other, but owing to the minuteness of the holes and to their conical shape the oil is retained. This quadran-

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gular column is itself surrounded by a double envelope made either totally of glass, or having two faces at least of that material, and in which an arrangement of partitions causes a current of air to flow against one of the perforated zinc faces and to force its way through the opposite. This current should not be too strong, but yet strong enough to constantly renew the air in contact with the oil.

The oil at last falls into a steam-jacketed reservoir, A, by means of which it can be heated to a temperature between 100° and 170° C. (212° to 338° F.). Finally, by means of a pump similar to Fig. 22, F, the oil thus heated is propelled into the reservoir at the top of the column, through the openings of which it again passes to be submitted afresh to the action of the air. An automatic arrangement enables the flow of oil to be stopped should the pressure inside the column become too strong. Raw oil, to which an appropriate drier has been added at the start, is that which is generally treated with this apparatus. Sugar of lead to the extent of 5 to 10 per cent. is the most suitable drier. However, oil previously boiled with manganese or litharge may also be treated, but air is propelled in this case at a lower temperature, say, 100° C. (212° F.); nevertheless, in spite of this precaution, the resultant oil is darker than when raw oil is used. Another arrangement by Walton consists in boiling the oil in large open pans; the hot oil is led into a chamber maintained at a suitable temperature by steam pipes. Here the oil which falls on the floor of the chamber is beaten with armed agitators, bringing it into the form of a spray which meets a current of air passing in the opposite direction; the oil falls back again into the chamber, which may be covered with glass, so that the action of sunlight may intervene.

Storer's Process.—Instead of *blowing* air into the oil, Dr. John Storer draws it in from the atmosphere by suction by the creation of a vortex current generated by a sort of screw propeller. By a mechanical arrangement the oil subjected to the vortex action flows into another compartment and returns to the surface to expel the vitiated air and to be again and again subjected to the same treatment. The screw propeller makes about 1,500 revolutions a minute. It will readily be seen that by the commingling of continually renewed fresh air, drier and oil are affected in a most complete and perfect manner by this process. The plant differs from Vincent's in the fact that the fumes are carried away, not by a dome riveted to the pan and forming part of it, but by a hood suspended over the pan, thus allowing fresh air to be continually brought in contact with the oil without the necessity of pumping. The atmosphere above the pan is therefore always freer from decomposition products.

Solidification of Boiled Oil—Linoleum Manufacture.—When boiled oil is *completely* oxidised it is insoluble not only in ordinary boiled oil itself, but also in the whole of the various solvents used as thinners, consequently manufacturers generally are content with a partially oxidised product which will dissolve therein.

But nowadays there is a demand for completely oxidised and

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consequently solid oils, which are incorporated by mixing and grinding with a great number of substances, to which they impart impermeability, elasticity, as well as insolubility, not only in the various solvents for oil but also in acids. This is the case in the manufacture of linoleum, for example, and other similar substances formed by a mixture of solid linseed oil, powdered cork, sawdust and rosin. The process most generally adopted consists in spreading in a very thin layer boiled oil, or oil thickened after one of the processes described, so that it may be completely oxidised; if the layer be too thick a skin is formed on the surface when oxidation verges completion, which stops all further oxidation from going on underneath, and complete solidification is prevented. When large quantities of solid oil are required it is necessary to have enormous surfaces at disposal, so that the layer of oil to be oxidised may be as thin as possible. Two methods are in vogue: (1) Linen cloths are passed through a bath of the oil to be oxidised, and thus become impregnated with the oil, and are afterwards suspended vertically so that the excess of oil drops off. When the layer of oil is dry the linen is passed through the bath once more and thus becomes charged afresh with oil, and again vertically exposed to the action of the air, and so on until a succession of solidified layers gives the thickness desired; (2) the following is a more simple arrangement. Oil is made to fall upon several thousand square yards of linen suspended vertically. The oil being in a very thin layer absorbs the oxygen necessary to change it to the solid condition very rapidly. This absorption is facilitated by working in warm chambers. The sequence of operations which follow each other, almost automatically produces, very cheaply, a superposition of perfectly dry pellicles, amalgamated together to form a mass of considerable thickness. To separate the solid oil from the linen, the latter is passed between two rolls heated by steam, and so adjusted that only the linen can pass through. In starting all that has to be done is to free the linen from solidified oil for about an inch, and feed it in between the rolls; these catch hold of the linen and draw it forward, leaving the solid oil behind. The linen may thus be used over again to prepare a fresh batch of solid oil. But when complex substances are to be made from the solid oil, manufacturers do not trouble to separate the two, the more so as the linen does not last long. In that case the linen charged with oil is ground up and mixed with the other ingredients.

Livache tried to use this oxidised oil by itself. When triturated with the majority of volatile solvents, spirits of turpentine, benzine, alcohol, ether, bisulphide of carbon, etc., it swells, becomes transparent and then divides into pieces of extreme tenuity, and thus forms a paste which may be spread out in a more or less thick layer. When the solvent has evaporated, an elastic homogeneous surface of oxidised oil is obtained, perfectly dry and of appreciable thickness. These pastes of oxidised oil may be combined with caoutchouc pastes, or with solutions of india-rubber and other substances, soluble in the

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solvents used, so as to obtain after evaporation a homogeneous product of peculiar properties partaking of those of the different substances entering into its composition.

The oil treated in the manner described until the maximum amount of oxygen has been absorbed, forms a gelatinous mass denser than water which can be drawn into strings. It is insoluble in alcohol, ether, spirits of turpentine, chloroform and carbon disulphide. When mixed with cork dust and rosin, "fillers," etc., it is rolled into linoleum. The highly-complicated machinery for inlaid linoleum, etc., is beyond the scope of this work. The following elementary analyses are by Rowland Williams :—

ELEMENTARY ANALYSES OF OXIDISED OILS FOR LINOLEUM MANUFACTURE.

	Raw Linseed.		Oxidised Linseed Oil for Linoleum Manufacture.						
	1	2	3	4	5	6	7	8	9
	%	%	%	%	%	%	%	%	%
Carbon -	75·03	75·40	74·32	69·74	69·52	64·74	65·40	68·64	64·38
Hydrogen -	10·78	10·64	10·04	9·57	9·49	9·01	9·00	9·24	9·01
Oxygen -	14·19	13·96	15·64	20·69	20·99	26·25	25·60	22·12	26·61

Reh's Process.—Good clarified Russian linseed oil is boiled with nitric acid of known strength to decompose the glycerine and oxidise the oil. The oil treated in this way is pumped into a tank waggon which sprinkles it on to a cloth which is oxidised by hot air.

Herkhorn's Patent.—In this process the alkaline earths and their soaps are added to the oil during boiling and oxidation, and a velvet lustre is thereby imparted to the mass. Rosin and rosinate of magnesia are added to the oil.

Pinette analysed three samples of linoleum with the following results. His method of working is so obvious as to require no description.

ANALYSES OF LINOLEUM.

	1	2	3
Water - - - - - - - - - -	3·39	3·01	3·41
Linseed Oil Soluble in Ether - - - - -	11·43	10·60	19·58
Linseed Oil Insoluble in Ether (Cork, Oxidised Oil)	77·24	73·63	54·16
Silica - - - - - - - - - -	2·94	3·99	4·31
Ferric Oxide - - - - - - - - -	1·78	1·79	8·86
Alumina - - - - - - - - - -	1·91	4·94	0·61
Lime - - - - - - - - - -	—	—	6·17
Alkalies - - - - - - - - - -	1·31	2·04	2·90
	100·00	100·00	100·00

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CHAPTER IV.

PRINTING INK MANUFACTURE.

Printing and Lithographic Ink.—The chief ingredient of printing ink is boiled oil, most generally linseed, sometimes hemp-seed, which is cheaper, but has a bad smell. No attempt is made to produce a fluid quick-drying oil like that used in the manufacture of oil varnishes, *viz.*, by boiling the oil to a temperature not exceeding 220° C. On the contrary, we have to produce a partially resinified, partially decomposed oil which will leave no greasy stain on paper by heating the oil above 250° C.

Refining Oil for Printing Ink.—The old tanked oil used should be of prime quality, so as to avoid the disagreeable smell and too deep colour of ordinary oil. It ought to be tanked for at least a year or two, so that all solid impurities may have completely separated out. In other words, what is known as old tanked oil should be used, but it ought still to undergo a special refining with sulphuric acid. Accordingly the oil is mixed with 1 or 2 per cent. of sulphuric acid of about 103° Tw., sp. gr. 1·515, and heated for several hours by means of a steam coil to a temperature of 90° C., taking care not to go beyond 100° C. The oil is then pumped into another tank containing hot water, with which it is well washed, running off the wash water and agitating afresh, and so on until all trace of acid is removed. The oil may then be boiled. As drying oils contain a certain quantity of fatty matter (palmitin) which does not dry, and causes the printing ink to blot or blur, it has been recommended to eliminate it by adding to every 1,000 parts of oil 1 or 2 parts of fuming nitric acid, which decomposes the palmitin into glycerine and palmitic acid, which may thus be precipitated as insoluble palmitate of lead or manganese. This process seems, however, to present many difficulties, which have prevented it from being adopted to any extent. Moreover, fuming nitric acid, in consequence of being easily decomposed, is by itself a dangerous substance; this danger is intensified to the highest degree when the acid is mixed with organic matter. Dangerous explosions may result and the oil take fire.

Description of Pan.—The oil is boiled either in a cylindrical copper pan, the lower half of which fits into the brickwork of the furnace, where it is kept in position by a collar which at the same time acts as a gutter to collect any overflow, or in a cast-iron

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enamelled pan. The pan is manipulated with a chain or by an iron band encircling its top, and attached to which are strong arms, by means of which it can be moved with safety. The capacity is 60 to 85 gallons. But in certain important factories the boiling is done in jacketed pans which do not cube less than 600 to 700 gallons. In such cases a mechanical agitator is kept going during the boiling process, which may last two or three days. (See Figs. 18-20.)

Boiling Process.—The pan is filled to half or at most three-quarters of its capacity, and a moderate heat applied so as to get quit of all trace of moisture; when the boiling goes on quietly the fire is quickened until volatile, pungent and disagreeable smelling products are given off. This takes place at a temperature of 250° to 270° C. (482° to 518° F.). In certain factories the workman satisfies himself as to this being the case by dipping in organic matter, such as a feather, for instance, which ought to inflame; or by projecting fine drops of water on to the surface of the oil, which as they fall decompose and scintillate, or, in other words, present, in consequence of the absorption of oxygen by the oil and the inflammation of the hydrogen, the appearance of stars. The fire is now carefully watched, for the oil may froth over or the vapours ignite. The frothing over is parried by adding a little cold oil. The oil becomes bright and more fluid at first, but, on being kept at this temperature for one or two hours, a brown red skin forms on the surface, which is skimmed off as rapidly as possible, because it is this skin which colours the oil brown. The temperature is now raised to 310° C., and kept at that for half an hour to an hour, watching the process with the greatest of care, as the vapours are very apt to catch fire. A well-fitting lid and wet cloths ought therefore to be always at hand. The fire is now allowed to die down, so that the temperature may descend to 260° to 270° C., when the pan is withdrawn from the fire. It is covered with a lid, and let stand to cool. Certain makers, instead of cooling the oil after withdrawing it from the furnace, set fire to the fumes, and let them burn for about five minutes—an operation they regard as indispensable in the production of a good article. Others contend that the same result is got by boiling the oil a little longer, which saves the appreciable loss of oil incurred by burning, and a much paler oil is obtained.

"String" Test.—The exact moment when the pan ought to be removed from the fire is determined by the "string" test. A small sample of oil is taken from the pan on an iron spatula, and is cooled rapidly by stirring it. A drop is taken and pressed between the finger and thumb, which are then gradually drawn apart as far as possible. The thread joining the finger and thumb together should stretch 1 to 2 inches without breaking, otherwise the oil is not sufficiently boiled.

Properties of Good Printing Ink.—Good printing ink has the requisite body of a pale, bright yellow colour—not deep yellow or brown—and is not cloudy from matters in suspension. It is very

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evident that the body of the oil will depend upon the purpose for which it is intended. For printing books and lithographic work an oil having a good body is required, whilst for newspaper printing a soft, almost fluid oil is used. According to Villon, who made an elaborate study of printing ink, five kinds of oil of different body or consistency are in the market, *viz.* :—

(1) Extra strong, (2) strong, (3) medium and (4) weak-bodied oils, and (5) drying oil. The strong-bodied oil (2) is used in summer for very particular work with the hand press. The medium oil (3) is used in ordinary weather. The weak-bodied oil (4) is used in winter with the mechanical press, whilst the drying oil (5) is used to prevent the formation of the oily ring around the letters.

Driers and Extra Strong-Bodied Oil.—Usually no driers are used, *because oil boiled with driers would clog the type*, and could not be removed even when washed with a hot alkaline solution. There is sometimes, however, an advantage in using such an oil, and to meet this want the oils known as "extra strong" and "drying" oils are manufactured.

The "extra strong-bodied" oil is made by boiling with lead salts, and the "drying" oil by boiling with an oxide or salt of manganese.

Strong-bodied oil (2) (*huile forte*) is prepared by setting fire to the oil, letting it burn for five minutes, and stirring several times. The flames are put out and the oil again set on fire, twice at least, and sometimes four times. The cooled oil strings like treacle. The "fatty" oil is made by shortening the burning process, and is intermediate between the other two. Nowadays a process similar to that used in making ordinary printing ink is adopted. The removal of grease by onions or bread is then effected by nitric acid, as previously indicated, or by magnesia. This latter, which is extensively used in England, consists in adding 2 per cent. of magnesia, boiling for an hour, and decanting. The oil is then run into a pan capable of holding 42 gallons, and gradually heated to 182° C. (327·6° F.), three hours being occupied in attaining this point. About 1½ lb. of finely ground bichromate of potash is then added, and the temperature is raised to 300° C. (572° F.), stopping the frothing of the oil by adding a little cold oil, then it is kept for half an hour at a temperature between 315° and 320° C. (599° to 608° F.), after 1½ lb. of Prussian blue have been slowly added. "Strong"-bodied oil is thus obtained. To make "fatty" oil, the oil is only heated for five minutes to 300° to 305° C. (572° to 581° F.). With "pale" oil the heat does not rise above 275° C. (527° F.), which is maintained a quarter to half an hour, according to the object in view.

The "extra strong-bodied" is added to the "strong-bodied" oil. The former is made by heating to 150° C. for an hour.

	Lb.
Linseed oil - - - - - - - - - -	200
Litharge - - - - - - - - - -	5
Umber - - - - - - - - - -	4
White lead - - - - - - - - - -	2

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The drying property of this boiled oil is tested by spreading it on a glass plate, when it should be perfectly dry next day, with neither air-bells nor dulness. If it be dull 5 per cent. of spirits of turpentine must be added.

The "drying oil" is obtained by heating for fifteen hours at 275° C. oil to which 0·05 per cent. of the hydrated oxide of manganese has been added, or by boiling oil for twenty-four hours at 300° C. (572° F.), with 5 per cent. of black oxide of manganese. If borate of manganese be used 4 lb. of this salt are beaten up with 25 lb. of linseed oil, and run into 2,000 lb. of linseed oil, and the whole heated for half an hour at 250° C. (482° F.), or a quarter of an hour at 316° C. (620·8° F.). Some manufacturers add 2½ per cent. of oxalate of manganese, whilst applying heat for three hours at 150° to 160° C. (302° to 320° F.), or simply add a manganese soap (manganese linoleate) previously dissolved in coal-tar naphtha, petroleum ether, or spirits of turpentine.

Use of Rosin.—When great body is required, without having resource to prolonged boiling, rosin may be added. Rosin is, in fact, extensively used in the making of printing ink, forming with the oil a mixture not unlike Canada balsam in appearance and consistency, and the latter itself is one of the best vehicles that can be used. With ordinary black ink common rosin is used, but for fine, coloured inks pale American rosin is employed. The rosin is melted, filtered to eliminate rubbish, and added to the oil during boiling. When very high-class inks are required for jobs to be executed in an especially neat and delicate manner, balsam of Peru, Canada balsam, or copaiba balsam are used instead of rosin. Copaiba balsam is said to produce a good extemporaneous ink when mixed with the proper quantity of soap and pigment.

Use of Soap.—Finally, a certain quantity of soap is added to the oil, which imparts suppleness, but its principal object is to facilitate the cleansing of the type, enabling this to be done with a brush. The soap ought to be perfectly dry, that is, to have been left for a long time in thin slices at 100° C. (212° F.), until it ceases to lose weight. Ordinary yellow rosin soap is used for common black ink, but for fine inks, for light and delicate shades of colour, white curd soap is used. Too large a proportion of soap retards drying and gives rise to irregular printing. The printing-ink maker should only use as much soap and no more as will cause the ink to work clean and without clogging the type.

Blue Black Ink.—A little indigo blue is also added to the hot oil to impart lustre, or 1 per cent. of Prussian blue may be added as the oil commences to boil, which, as it dissolves, yields a beautiful blue black.

Manufacture Proper.—Generally in the actual manufacture of the ink, the boiled oil is moderately heated with the simple addition of rosin and soap alone until the mass becomes tranquil and homogeneous; the colour is then added and ground in the mill until

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perfectly cold. Another process consists in melting the rosin until destructive distillation commences, then to add soap in slices, and then the boiled oil. Heat is now applied more strongly, so as to increase the fluidity of the mass, then the colour is added, and the whole run into a receiver, from whence it passes to the mill while still hot. The nature and the quantity of the colouring matter depend upon the consistency of the oil and the object in view, but heavy, dense, mineral colours should, as far as possible, be excluded, using in preference light colours, which give a more homogeneous and impalpable ink.

Thus the following colours are habitually used, *viz.*, for

Black—Lamp-black. This pigment may be used alone or with an equal weight of Prussian blue for the purpose of removing the brown tone of certain lamp-blacks. Bone black is too dense to be used by itself. It is, however, added in small quantities to lamp-black so as to produce an ink for printing wood engravings. Carbon gas black is a magnificent colour.

Red—Lake, cochineal, carmine, aniline lakes; very rarely red lead or vermilion. Vermilionette, containing 90 to 98 per cent. of a mixture of red lead and barytes, is too heavy and dense.

Lakes made with tannic acid go leathery in oil.

Blue—Prussian blue or indigo; a rich tone is got by grinding a little Indian red, Fe_2O_3, with the Prussian blue and indigo; ultramarine is inadmissible as it is easily bleached.

Yellow—Yellow ochre or chrome yellow, chinoline and other aniline lakes.

Green—Mixtures of blue and yellow.

Brown—Sienna and umber.

The printer or printing-ink maker could not do better than consult Jennison, *Lake Pigments* (Scott, Greenwood & Co.). Samples of colour printing produced by lakes of all colours and shades of colour are given therein, with instructions how to make them.

Characteristics of Good Printing Ink.

Printing ink ought to satisfy several tests. (*a*) It ought to be perfectly homogeneous, for the least particle of extraneous matter (grit, etc.) will cause a blot in the printing; (*b*) it should not alter when exposed to the air in bulk for a long time, but when spread in a thin layer it should dry very quickly, without, however, commencing to do so on the type, from which it should be easily removed by washing; (*c*) the colour of dry printing ink should be rich and brilliant; (*d*) it should not penetrate the paper too deeply but deep enough to prevent it being detached from the surface; (*e*) finally, it ought to have no bad smell.

The proportions of different substances entering into the composition of printing ink vary much. They may, however, be reduced to three types :—

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	Best. Lb.	Medium. Lb.	Ordinary. Lb.
Boiled oil	100	100	100
Rosin	25	50	75
Soap	4	6	8
Drying oil	6	9	12

For ordinary, such as newspaper, work great economy is necessary, hence in place of boiled oil there has been used a mixture of raw oil and thickened turpentine; the quantity of rosin is increased and the requisite fluidity obtained by the addition of rosin oil.

The proportions given by Andés are as follows, according to the amount of body required:—

	Lb.	Lb.	Lb.
Raw linseed oil	140	110	90
Rosin oil	240	240	240
Rosin	210	210	210
Rosin (*i.e.*, yellow) soap	5	5	5
Thickened turpentine	5	5	5

Inferior inks are made very cheaply by replacing linseed oil by rosin oil, for which purpose the following recipes have been given:—

	Lb.	Lb.
Rosin oil	1,000	1,000
Rosin	400	780
Rosin soap	100	780
Ordinary soap	100	180

GOYNEAU'S PRINTING INK.

	lb.	lb.	lb.
Linseed Oil	979	400	980
Litharge	125	60	122
Rosin	735	380	980
Syrup	245	980	958

	I.	II.
	lb.	lb.
1. Venice Turpentine	60	9
2. Olein	30	4
3. Soft Soap	80	10
4. Lamp-black	50	4 or more
5. Prussian Blue	40	—
6. Oleic Acid	20	—
7. Water	20	—

Mix 1, 2 and 3 with aid of heat. Stir in 4 after sifting finely, then incorporate with I. the solution of 5, 6, 7. The 40 lb. of Prussian blue may be replaced by 20 of indigo carmine first rubbed up with water. It is claimed for II. that it can be removed from old printed paper and renders printers' lye unnecessary.

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	Common Black.	Rotary Soft.	Jobbing.
	lb.	lb.	lb.
Paraffin Oil - -	25	25	25
Rosin - - - -	45	40	—
Lamp-black - -	15	15	15
Dammar - - -	—	—	45

Rosin of different grades may be used or Burgundy pitch. The paraffin oil may be replaced by rosin; the proportion of lamp-black is adjusted accordingly. Other pigments in suitable proportion may be substituted for lamp-black.

GUNTHER'S PRINTING INK.

	Lb.	Lb.
Heavy tar oil - - - - - -	45	28
Pitch or asphaltum - - - - -	40	40
Rosin pitch soap - - - - - -	12	24
Fish oil - - - - - - -	5-8	—
Spirit soluble aniline dye in powder - -	3-15	8

The heavy tar oil is first acted on by chlorine at 212° F., or is heated with nitric acid to deodorise it. The oil is then boiled with 5-10 per cent. of chloride of copper, heated to remove free acid, and then dissolved in warm water or stirred into the oil to give the oil a brownish-black colour and thus reduce the aniline violet to a minimum—$\frac{1}{8}$ per cent. is then sufficient. The oil thus treated can be used as a stamping ink for post-office work without any addition.

PRINTERS' INKS FOR VARIOUS PURPOSES.

Use.	Rotary Machines.						Newspaper.		Book.			Illustration.		
	lb.	lb.	lb.	lb.	lb.	lb.	lb.	lb.	lb.	lb.	lb.	lb.	lb.	lb.
Vehicle - - -	70	72	72	72	74	74	78	76	77	79	80	78	78	78
Lamp-black - -	30	28	28	28	26	26	22	24	23	21	20	20	19	19
Prussian Blue - -	—	—	—	—	—	—	—	—	—	—	—	2	2	1
Indigo - - -	—	—	—	—	—	—	—	—	—	—	—	—	1	—
Chinese Blue - -	—	—	—	—	—	—	—	—	—	—	—	—	—	2

PRINTING INKS—VEHICLES.

	Thin.			Medium.			Thick.		
	lb.	lb.	lb.	lb.	lb.	lb.	lb.	lb.	lb.
Rosin - - - -	25	25	25	50	50	50	77	77	77
Boiled Oil - - -	100	100	100	100	100	100	100	100	100
Rosin Soap - - -	3	3	3	10	10	10	7	7	7
Weak-boiled Oil - -	7	4	—	9	6	—	12	9	—

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	Thin, with Boiled Oil.			Medium, with Boiled Oil.			Thick, with Boiled Oil.		
	lb.	lb.	lb.	lb.	lb.	lb.	lb.	lb.	lb.
Rosin Oil - - -	50	50	50	50	50	50	50	50	50
Rosin - - - -	25	50	75	50	50	50	50	50	50
Boiled Oil - - -	50	50	5	50	50	50	50	50	75
Rosin Soap - - -	3	5	7	3	5	7	3	5	7
Weak-boiled Oil - -	7	9	12	4	6	9	—	—	--

PRATT'S ORIGIN ROSIN OIL PRINTING INK VEHICLE.

	lb.	lb.
Rosin Oil - - - - - -	20	50
Rosin - - - - - -	8	39
Yellow Soap - - - -	2	9

	Thin.		Medium.		Thick.	
	lb.	lb.	lb.	lb.	lb.	lb.
Rosin Oil - - - -	96	95	240	80	240	80
Raw Linseed Oil - - -	84	—	210	—	210	—
Rosin Soap - - - -	2	7	5	70	5	7
Thick Turpentine - - -	2	—	5	—	5	—
Rosin - - - - -	—	100	—	100	—	100
Boiled Oil - - - -	52	35	1,000	3,500	87	25

Ink for Steel Engravings.

This oil, according to Villon, who made a special study of ink for steel engravings, is prepared like printing ink, the only difference being that it is boiled a little longer. Old tanked linseed oil, *i.e.*, well-aged oil, is the oil employed, or, better still, walnut oil; but this latter is never used alone, a little linseed oil, varying from 12 to 50 per cent., according to the quality desired, being always added to give it more viscosity. The boiling process, although an old one, is that still used. It consists in heating 5 gallons of oil in a pot until it takes fire or gives off white fumes, which are set on fire. It is left to burn for five minutes, taken off the fire and placed on a furnace filled with hot ashes, where it is let burn a few minutes longer. A piece of stale bread, held by a pair of pliers, is dipped into the oil until it reddens, without, however, charring; it is replaced by a second slice, and that by a third—the bread is sometimes replaced by peeled onions. Although no explanation is given of this process, it would appear to

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be necessary. At any rate practical men think so. The pot is now covered and allowed to cool. There is thus obtained a *bright*, greenish oil (*huile claire*), more consistent than the raw oil, but with no body.

De Luxe Illustrations, Ink for.—Villon gives the following proportions for ink to be used in the production of *de luxe* illustrations :—

	Lb.
Limpid oil (*huile claire*)	1,000
Strong-bodied oil (*huile forte*)	250
Lamp-black	500
Prussian blue	50
Rosin (yellow) soap	25

The Prussian blue is first added to the clear oil in a pan fitted with a mechanical agitator, and heated to 100° C. (212° F.) by a steam jacket. The heat is raised to 150° C. (302° F.), and the rosin soap is mixed with continual stirring. It is only when perfectly cold that the black is introduced into the mixture, then the *huile forte* (strong-bodied oil). The whole is then passed through rollers. Small quantities may be ground in a mortar worked by power. [By using carbon gas-black instead of "lamp"-black a superior jet black is obtained.]

Black Ink for Engraving Purposes.—Villon's researches resulted in his selection of the following formula as yielding a very black ink for engraving purposes, without reddish reflex, and of irreproachable composition, which keeps well, and each of the ingredients of which plays an important part :—

	Lb.
Huile claire (pale limpid oil)	1,000
Huile forte (strong-bodied oil)	200
Carnanba wax	25
Paraffin	35
Yellow soap (rosin)	25
Paris violet	0·5
Prussian blue	40
Cork black	100
Blood black	50
Winelees black	200
Ivory black	150

Numerous other substances are also used to impart consistency and unctuosity, such as Canada balsam, ozokerit, asphaltum, stearic acid. It is for each manufacturer to select those materials best adapted for the object in view. The retention of the copper lustre of Prussian blue in the matter printed from an ink made from that pigment is a subject which has interested and engaged the attention of not a few. Some claim to have succeeded by the use of lard, etc. But the copper bronze reflex of modern printed matter is undoubtedly due to nigrosine black and aniline blues. With the use of these pigments the difficulty is not in retaining the bronze, it is in eliminating it.

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PRINTING INK VEHICLES.—(LEWKOWITSCH.)

	Density at 15° C.	Free Acid Calculated as Oleic Acid.	Saponification Value, MgKHO.	Unsaponifiable Matter.	Oxidised Acids.	Iodine Value.
Raw Linseed Oil - -	0·9321	0·85	194·8	—	0·30	169·0
Tint - - - - -	0·9584	1·46	197·5	—	1·50	113·2
Thin - - - - -	0·9661	1·76	196·9	0·62	2·50	100·0
Middle - - - -	0·9721	1·71	197·5	0·85	4·20	91·6
Strong - - - -	0·9741	2·16	190·9	0·79	6·50	86·7
Extra Strong - - -	0·9780	2·51	188·9	0·91	7·50	83·5
Burnt Thin - - -	0·9675	6·93	195·5	1·35	0·85	92·7
Oxidised Oil (weak) - -	1·03	18-28·4	221	0·81	42·82	58·8
Oxidised Oil (strong) -	1·05	18·49-28·9	223·5	0·97	44·19	53·5
Dried Linseed Oil - -	—	12·67	171·5	0·81	31·58	93·9

PRINTING INK VEHICLES.—(LEWKOWITSCH.)

MIXED FATTY ACIDS.

	Density at 15° C.	Melting Point, Degrees C.	Solidifying Point, Degrees C.	Mean Combining Weight.	Saponification Value.	Iodine Value.
Raw Linseed Oil - -	0·923	24-26·5	—	286·5	195·8	145·6
Tint - - - - -	0·941	20·5	15	—	—	118·3
Thin - - - - -	0·949	22	18	—	—	108·8
Medium - - - -	0·950	24	22	272·6	205·8	97·7
Strong - - - -	0·953	25·5	24	270·1	207·7	87·3
Extra Strong - - -	0·955	27	23	269·8	207·9	90·8
Burnt Thin - - -	—	23	19	—	—	99·3
Oxidised Oil (weak) - -	—	28	26	241·4	232·4	63·2
Oxidised Oil (strong) -	—	27	25	242·5	231·3	60·6
Dried Linseed Oil - -	—	26	22	268·8	208·7	100·3

ADDENDA.

Lithographic Printing Ink Delicate Tints.—Venice turpentine, 5; castor oil, 15; white wax (bees or paraffin), 1 (all in lb.). Lemercier says Burgundy pitch is an essential ingredient, obviating great heat in boiling oil; result is paler, keeps unctuous, is more pliable; with moderate pressure the ink easily quits the stone for the paper, often entirely, but the greasy spots are inked again in due course.

Lithographic Crayons.—(*a*) Wax, 2; (*b*) tallow, 1½; (*c*) white soap, 6½; (*d*) shellac, 3; (*e*) lamp-black, 1½ (all in lb.). Melt (*a*) and (*b*), add (*c*) gradually, stirring constantly. Heat till mass fumes, take off fire, enflame for one minute at most, add (*e*) stirring, put on fire again, boil ¼ hour still stirring, cool slightly, run on a soap-smeared sheet of paper and mould into sticks.

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CHAPTER V.

RUBBER SUBSTITUTES.

India-Rubber Substitutes.[1]

Drying oils are used in the manufacture of solid elastic compounds which can be used as rubber substitutes. Sulphur, chloride of sulphur and nitric acid are the reagents used.

Imitation rubbers generally have drying oils as a basis, that is to say, linseed oil, walnut oil, etc. (this latter oil is too dear, and is only mentioned to show that the same results can be got with it as with linseed oil). But non-drying oils are also extensively used, *e.g.*, colza oil in Germany and maize oil in America, in the making of chloro-sulphuretted oil substitutes from sulphur chloride. Receipts are innumerable; each maker has his own sleight of hand; practice and experience play an important part, and an irreproachable imitation is not made on the first attempt. Numerous experiments are necessary to arrive at the right proportions and the proper temperature. We do not intend to enumerate all the known and proposed receipts for making imitation rubber. Even if we knew them all, one scruple would prevent us; generally the published receipts are obsolete, fit to guide the beginner in practical researches, but not sufficient for immediate and profitable use in trade. It will be sufficient, therefore, to dwell upon the origin of the two principal varieties of imitation rubbers, *viz.*, (1) oxidised oil and (2) vulcanised oil substitutes.

(1) *Oxidised Oil, Black Substitutes.—Sacc's Experiments on the Action of Nitric Acid on Linseed Oil.*—Sacc, whilst studying in 1846 the saponification of linseed oil by caustic soda, examined the action of nitric acid on that oil. When 100 parts of linseed oil and 200 parts of nitric acid, diluted with four times its volume of water, are gently heated with continual stirring, the oil will be seen to become of a brownish-red colour; there is abundant disengagement of nitrous vapours, the oil thickens, and at the end of four hours the mass acquires a very decided syrupy consistency.

Zonas' Method of Preparing "Caoutchouc des Huiles".—L. Zonas, resuming in 1848 the experiments of Sacc, set fire to linseed oil previously viscous, then, after having partially burnt it, he treated the residue with dilute nitric acid. This was the beginning of oxidised oil rubber substitutes (*caoutchouc des huiles*).

[1] See also the writer's translation of *India-rubber and Gutta-percha* (Scott, Greenwood & Co.).

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Sollier and Rattier's Attempts.—In 1854 F. Sollier, whether ignorant or not of these laboratory experiments, tried to prepare from linseed oil a product capable of replacing rubber in some of its essential applications. Rattier patented a similar product. *Present Process.*—A certain proportion of linseed oil is heated until it is converted into a brown viscous mass. To thus convert 10 kilogrammes (say 22 lb.) it is necessary to heat for at least twenty-four consecutive hours. The viscous mass is afterwards treated in the hot for a few hours with nitric acid until it has assumed a thick plastic consistency, and when cooled in the air becomes solid. The product is freed from the excess of acid by kneading it for some time in a rather weak alkaline lye until it no longer has an acid reaction. In the cold it exhibits the appearance of natural rubber; it is rather elastic, softens in hot water, and, unlike rubber, becomes plastic like gutta-percha. It is soluble in spirits of turpentine, carbon disulphide and alkalies. Acids precipitate it unchanged from its alkaline solution.

Although dilute nitric acid and linseed oil yield a product somewhat analogous to the one next to be described, *viz.*, that obtained by the action of chloride of sulphur on oils, yet in the latter case it would appear to be the sulphur which is the active agent, and which explains why we can have the same substance by using either drying or non-drying oils. In the present case, however, we are confronted with a rapid oxidation process exclusively confined to those oxidisable principles only found in drying oils. The oil is first heated so far as to render it viscous; it is then boiled for a long time with dilute nitric acid. A solid elastic, brown substance, which does not stick to the fingers, is obtained analogous to caoutchouc, whence its name of black artificial rubber. The same substance is obtained with the different drying oils, but in proportion to the intensity of their drying properties. Linseed oil and walnut oil yield eight to ten times as much as poppy-seed oil.

As soon as discovered the product was utilised for the manufacture of canvas, imitation leather for saddlery and carriage building, and travelling articles of a suppleness and fitness leaving nothing to be desired. Its use, although less and less considerable for some time back, is still in vogue, and if it be but rarely used alone it is still sometimes added to articles made from pure rubber. As it adheres perfectly to all fabrics without altering them or penetrating them too deeply the manufacturers of waterproof canvas often resort to it. It may also be applied without any difficulty to wood, to stone, and to metals, and in so doing it contracts a most remarkable adherence.

Properties of Oxidised Oil Rubber Substitute.—This substance does not melt, but may be differentiated in a very precise manner from the final product yielded by the complete oxidation of drying oils, *viz.*, *linoxin*, by the fact that it cannot be saponified by a concentrated *aqueous* solution of potash. On heating, an emulsion is obtained which does not separate on the addition of an acid. With *alcoholic* potash, on the contrary, complete saponification takes place,

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and the addition of an acid liberates a mixture of fatty acids of complex constitution. With carbon disulphide there is an emulsion; in petroleum the substance swells without dissolving; in a mixture of alcohol and ether it swells and dissolves if a large quantity of ether be added, whilst it is precipitated if alcohol be added in too great proportion; finally, it is soluble in ether and in a large excess of spirits of turpentine. Rubber substitute, prepared from linseed oil and nitric acid, would appear to consist on the one hand of a mixture of substances present in the original oil and not sensibly changed by the nitric acid treatment, and on the other hand of products oxidised by the nitric acid and transformed into linoxin, the relative proportions of these substances masking the individual properties of each of them.

This substance is used either in solution in ether or in spirits of turpentine in waterproofing fabrics, or is added directly in the solid state to india-rubber.[1]

(2) *Vulcanised Oils.—Chloride of Sulphur Process, White Substitutes.*—By mixing linseed oil with 5 per cent. of chloride of sulphur the oil becomes very thick, becoming plastic with 15 to 20 per cent., the solidity increasing as the percentage rises to 25 per cent. If the mixture be cooled solid sheets are obtained, on which other layers can be superimposed in such a manner as to obtain plates thick enough for making printing rolls, boxes, knife handles, etc. If a certain quantity of carbonate of lime be added the carbonic acid evolved converts the mixture into a white spongy mass, which is mixed with natural rubber. Chloride of sulphur has a similar action on other oils such as earthnut or colza.

Nickles and Rochleder were the first to observe the action of chloride of sulphur on fatty oils, by which they are transformed into a substance rather resembling rubber. Mixed with any vegetable oil chloride of sulphur immediately converts that oil, almost at the ordinary temperature, into a solid substance possessing great hardness at times.

Parkes' Patent.—Parkes, to whom industry owes the process of vulcanising rubber by chloride of sulphur, patented a process for vulcanising linseed and rape oils by means of chloride of sulphur (British Patent, 22nd October, 1855, No. 2359).

Roussin's Researches.—In 1858 Roussin communicated to the Academy of Science the result of his researches on the action of chloride of sulphur on oil (29th November). If we take 100 parts of linseed oil and about 25 parts of chloride of sulphur a compound is obtained possessing the maximum of hardness, but 100 parts of linseed oil and 15 to 20 parts of chloride yield a more supple product; and, finally, 100 parts of oil and 5 of chloride thicken the oil, it is true, but do not harden it. This latter combination is soluble in all ordinary

[1] *Synthesis of Genuine Rubber.*—The attempts that have been made to convert spirits of turpentine into real rubber have not as yet been carried out in practice.

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oils, which is not the case with thicker combinations, which only swell in these vehicles. When a certain quantity of linseed oil is diluted with 30 to 40 times its weight of carbon disulphide, and if one-fourth of the weight of oil be replaced by the same quantity of chloride of sulphur, a product is obtained which remains liquid for a few days. If this solution be applied on glass, wood, etc., the carbon disulphide evaporates immediately, and a coat of varnish is soon obtained. Several precautions must be taken in order to produce mixtures of chloride of sulphur and oil possessing the properties just referred to.

It is first of all necessary to select a chloride of sulphur containing the strongest possible proportion of sulphur. This product, which is liquid, is poured rapidly into the oil, and the mixture is agitated in order to obtain a uniform mass. Soon the oil heats, the reaction is finished, and the oil hardens or forms a soft compound, according to the proportions of chloride. It is essential to only operate on small quantities at a time, and to avoid such an elevation of temperature as would volatilise the chloride, produce bubbles, and even blacken or carbonise the oil. *Sulphur dichloride* should never be used in preparations of this kind; its action is too strong and too rapid; the oil being treated would carbonise very rapidly and the preparation would be irremediably burnt. When the two substances are intimately mixed the product is run on to a glass plate, or upon another plane polished surface, where it is equalised, then after the lapse of five to ten minutes, according to the temperature, whether greater or less, combination is complete.

One hundred pounds of the oil are mixed with 4 gallons of benzoline, and there is added a mixture of 25 lb. of sulphur chloride in 2 gallons of benzoline. The work should be done in a closed vessel provided with a stirrer, and the chloride of sulphur should be added only in small quantities at a time. Some heat is generated which causes the petroleum spirit to vaporise, while a little gentle heat at the end is sufficient to drive off the remainder. These chloride of sulphur substitutes are generally of a pale yellowish colour, rather spongy in texture. They contain but little free oil, and no free sulphur. They work with the rubber better than do the oxidised substitutes previously described.

As a final result a pellicle is obtained, which it is easy to raise; all that is required is to detach one of the corners with the point of a knife and to pull the remainder gently by means of this corner. Moreover, several of these layers may be superimposed, taking care so that they may amalgamate together well, to apply one above the other when the latter is cold. In order to ensure perfect amalgamation, moisture must be avoided, which decomposes the chloride and prevents adherence.

By working as just described, solid plates are obtained capable of being used in making numerous articles which could only be done previously with rubber. These articles are perfectly transparent,

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provided care has been taken after making them to keep them in an oven, or in a hot chamber, for a sufficiently long time for the vapours disengaged by the chloride to escape. They resist atmospheric influences, acids and weak alkalies; but they are brittle, and emit a peculiar odour, from which it is difficult to free them.

All vegetable oils may be used in the making of these substitutes, but linseed, rape-seed, earthnut and colza oils are preferred.

Brown Substitutes—Oils Vulcanised by Flowers of Sulphur.—Rubber substitutes made from chloride of sulphur, as we have just seen, are colourless, and in texture in no way resemble commercial rubber. Their manufacture is also delicate, and liable to very frequent failures, due principally to the too energetic action of the chlorides upon the oils in presence of however slightly elevated a temperature. It has been found possible to overcome this difficulty by the direct vulcanisation of linseed oil, by flowers of sulphur, which produces a black substance approaching more nearly to the natural colour of rubber, and which, by its slower and more gradual elaboration, avoids the innumerable accidents of a reaction accomplished too rapidly. This substitute, which at the present day has in the greatest number of instances displaced oil vulcanised by chloride of sulphur, is prepared as follows: Linseed oil previously heated to a temperature of 100° C. is intimately mixed with 5 to 10 per cent. of flowers of sulphur, according to the object desired, then heated gradually to a temperature of about 130° C. (266° F.). The mixture rapidly turns brown, and when it has got to the desired temperature and acquired a very pronounced syrupy consistency, it is left to itself, without, however, allowing the temperature to lower below 100° C. (212° F.). Vulcanisation is known to be finished by the deep brown, almost black, colour of the mass, and its ever-increasing thickness. At this point the process is conducted in the same way as in the case of chloride of sulphur substitutes. That is to say, the vessels are emptied on smooth, cold surfaces, so as to render the product detachable after complete cooling. One method of making substitutes from non-drying oils consists in taking 100 lb. of good Stettin colza oil and mixing it with 15 lb. flowers of sulphur, and heating the mixture with frequent stirring to a temperature of about 300° F. until a dark-coloured, almost solid, mass is obtained. On cooling, the substitute is of a rubber-like character, but devoid of the elasticity and tenacity characteristic of rubber. During the process, part of the sulphur enters into combination with the oil, part remains free. It is desirable that the free sulphur should be very small. The average amount is 2·5 per cent.; when the amount reaches 5, it becomes objectionable, as it tends to produce defective goods.

All the oil should be combined with the sulphur, as any free oil is found to act upon the real rubber with which the substitute is mixed, and causes it to decay. The fatty compound formed by the interaction of the oil and the sulphur is not soluble in petroleum ether or petroleum spirit, but is acted on by alcoholic solutions of caustic soda.

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The substitutes of which we have just spoken are sometimes employed alone in the manufacture of waterproof cloth, water-pipes, etc., sometimes in combination with natural supple rubber for all other industrial uses.

At the present day, therefore, it would appear that only one class of organic compounds plays an important *rôle* in the rubber industry, *viz.*, the products sold under the name of rubber substitutes, artificial rubber or imitation rubber, made by heating oils, whether drying or non-drying, with sulphur or chloride of sulphur, and the non-sulphuretted substitutes made by oxidising linseed oil.

Analysis.—Substitutes are generally met with in the form of yellow or brown elastic masses, without cohesion, breaking up under pressure, greasy and moist to the touch. Two of these substances gave the following results :—

	I.	II.
Water	1·00	0·85
Sulphur	6·17	6·4
Ash	5·52	0·8

Substitutes in fact behave towards solvents like rubber itself ; insoluble in alcohol, they only dissolve with difficulty and incompletely in benzol, carbon disulphide and spirits of turpentine, etc. To detect the presence of oils or fats in manufactured rubber, a method has been proposed which yields, in experienced hands, useful results. The rubber is digested in carbon disulphide to which 5 per cent. of spirits of turpentine has been added, the solution is filtered after a few hours, and distilled. A notable residue indicates the presence of foreign bodies of a fatty nature. The method has several drawbacks : first, vulcanised rubber is slightly soluble in the mixture of carbon disulphide and alcohol ; the experiment is not conclusive unless the fats are present in notable quantity ; finally, sulphur is likewise dissolved, and may give rise to error. In spite of these drawbacks, from a qualitative point of view, the method, applied with discretion, may yield useful indications. For a quantitative estimation the process cannot be adopted, because the substitutes only dissolve partially, even when isolated and repeatedly digested in alcoholised carbon disulphide. The sorts examined, in dissolving, left a residue of 20 to 30 per cent. of their weight, and on each treatment afterwards still further lost 1 to 2 per cent., so that it cannot be admitted that the substitute is insoluble, and that it is the proportion of unchanged oil or fat which alone dissolves. Substitutes dissolve completely in petroleum at a high temperature, as vulcanised rubber does itself. Ligroin only dissolves them partially. Aqueous soda dissolves them with difficulty and incompletely. The action of alcoholic soda is dealt with further on.

Hübl's iodine addition method seemed likely to yield indications in its way, for rubber hardly absorbs iodine, whilst the sulphuretted oils should readily fix iodine, like the oxidised oils which almost retain their primitive iodine value. Preliminary experiment led to this

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unexpected result: *sulphurétted oils do not absorb iodine and behave like quasi-saturated compounds.*

Henriques tried to separate the sulphur from the substitutes and to isolate and weigh the regenerated fatty acids. He treated the substitutes with alcoholic soda, to which different salts which ought to absorb sulphur were added—salts of lead, mercury, copper and zinc; but in whatever way operated on, the fatty acids, isolated from the alcoholic lye in very variable quantity, always contained equally variable quantities of sulphur. He tried, without any better success, to effect saponification and desulphurisation in a closed vessel at a high temperature, by replacing ethylic alcohol by amylic alcohol. However, it was found by these experiments that substitutes dissolve freely and completely in alcoholic soda, and on this property may be based at least an approximate method of analysis. In this field sharp and complete separations such as can, for example, be done in inorganic analysis are quite impracticable. Results can only be approximate. We have in fact to deal not with simple combinations, but with very complex mixtures, like rubber itself, which may contain substances belonging to different classes of bodies. The following experiment shows that substitutes, or at least their organic constituents, are completely soluble in alcoholic soda.

One gramme of substitute was boiled in a flask attached to a reflux condenser, with an excess of caustic soda (7 to 8 per cent. Na_2O). After a few hours the alcohol was distilled off, the residue dissolved in water, and filtered through a tared filter.

Weight of the dry residue - - -	0·041 = 4·1 per cent.
Weight of the ash - - - -	0·0413

The residue, therefore, no longer contained any trace of organic matter. Another substitute which left no ash dissolved without residue.

Both white and brown substitutes present themselves under the form of slight yellowish, clotted, elastic masses, with a neutral reaction and a slightly penetrating oleaginous odour. Water extracts nothing; acids and alkalies but little; neither do the majority of neutral organic solvents.

The characteristic of these products is their high percentage of chlorine, almost as high as their percentage of sulphur. According to the behaviour of the products with solvents, the chlorine should exist in organic combination. If, therefore, as all the facts lead us to suppose, and as the experiments which will be detailed further on show, the substitutes examined result from the action of chloride of sulphur upon oils, that reagent has entered entirely, chlorine and sulphur, into the molecule of the proximate constituents of the oil. In order to facilitate the explanation of the results, Henriques' analyses are given below.

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I. ANALYSES OF COMMERCIAL RUBBER SUBSTITUTES.

	White Substitutes.			Brown Substitutes.	
	A.	B.	C.	A.	B.
Sulphur in the substitute	6·4	6·17	8·25	15·48	17·71
Chlorine in the substitute	5·0	5·86	8·88	0·7	0·36
Water in the substitute	0·85	1·0	—	—	—
Ash in the substitute	0·8	5·51	—	—	—
Percentage of fatty acids	90·45	73·58	—	—	—
Sulphur in the fatty acids	6·12	6·45	8·15	14·14	15·20
Chlorine in the fatty acids	0·83	0·43	—	—	—
Iodine value of the substitute	30·9	31·0	32·6	42·0	42·0
Iodine value of the fatty acids	91·3	91·2	102·3	129·0	125·6

II. ANALYSES OF SUBSTITUTES PREPARED WITH—

	A.	B.	C.	D.	E.	F.	G.	H.	I.
Sulphur in the substitute	9·34	4·78	8·28	6·59	7·68	—	4·82	10·6	6·23
Chlorine in the substitute	8·84	4·85	7·62	5·95	7·44	—	6·70	8·95	5·36
Water in the substitute	3·02	0·85	—	—	—	—	—	—	—
Ash in the substitute	0·0	0·0	0·0	0·0	0·0	0·0	0·0	0·0	0·0
Percentage of fatty acids	79·6	1·67	86·89	87·95	74·90	—	85·35	—	—
Sulphur in these acids	9·88	4·06	8·34	6·54	8·32	—	5·32	—	6·44
Chlorine in these acids	Traces.	0·60	Little.	Little.	—	—	0·26	Traces.	Traces.
Iodine value of the substitute	56·3	52·6	32·5	29·9	33·6	42·8	35·2	21·9	30·3
Iodine value of the acids	160·3	141·2[1]	101·5	102·8	133·3	129·2	136·2[2]	143·5	91·5
Acetyl value	21·0	19·6	31·0	—	—	—	—	105·6	51·3

A, Raw linseed oil (fresh). B, Oxidised oil. C, Rape oil (fresh). D, Oxidised rape oil. E, Oxidised poppy-seed oil. F, Mixture of oxidised linseed and poppy oils. G, Castor oil with a minimum dose of chloride of sulphur. H, Castor oil with a maximum dose of chloride of sulphur. I, the oil termed soluble castor (oxidised cotton-seed oil).

The determination of the sulphur of substitutes necessitates the same precautions as rubber. Oxidation by nitric acid, followed by fusion with an alkaline oxidising agent, alone yields concordant results. In order to estimate the chlorine, nitrate of silver was added to the nitric acid, so as to avoid all loss by volatilisation of the hydrochloric acid. After alkaline fusion, the whole is digested in water, the insoluble silver combinations are separated (generally metallic silver), and the sulphur is estimated in one portion of the liquid as sulphate of baryta, and the chlorine by titration with nitrate of silver and sulphocyanate. As already mentioned, oils solidified by chloride of sulphur only absorb insignificant quantities of iodine. Sample A, Table I., for example, gave an iodine value, according to Hübl, of 7·2. However, this value is only apparent. The feeble

[1] Another determination gave iodine value = 121·0.
[2] Two other determinations gave 147 and 152·1.

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iodine absorption is partly due to the fact that the product is almost insoluble in chloroform. By frequently agitating the finely divided substitute in suspension in that liquid with an excess of iodine solution, and leaving it in contact for twelve hours, Henriques obtained in the case of the sorts A and B, Table I., iodine values of 30·9 and 31. Compared with the iodine values of the drying oils used to make these substitutes, these figures are very low. Chloride of sulphur seems to partially saturate the free valencies of the oil to as great an extent by its chlorine as by its sulphur, possibly by its chlorine alone. It is to be noted that substitutes treated with iodine, in chloroformic solution, strongly retain the metalloid, and it is necessary in the back titration by hyposulphite to insist on stirring energetically and for a long time so as to destroy all the iodine in excess.

It should be interesting to observe how substitutes behave on saponification. We already know that they are completely soluble in alcoholic soda. In this reaction, the chlorine is almost eliminated, whilst the percentage of sulphur in the fatty acids corresponds exactly with the percentage of sulphur in the substitute. However, the proportion of fatty acids found is always lower than simple saponification would imply, even taking into account the elimination of the chlorine. Thus, Sample A gave 90 per cent., Sample B only 79·4 per cent. of fatty acids. A portion of the oil and a corresponding quantity of sulphur must therefore have undergone a transformation of a different order. The liquid from the saponification contains much chlorine but no apparent sulphur nor sulphuric acid, sulphuretted hydrogen nor sulphurous acid. But on evaporating these liquids in presence of the excess of hydrochloric acid added to liberate the fatty acids to the point where hydrochloric acid fumes begin to be given off, the presence of much sulphuric acid is demonstrated. It would seem, therefore, that the chloride of sulphur, with the assistance of oxygen borrowed either from the air or the substitute itself, had transformed a portion of the oil into a sulpho-oleic acid analogous to those produced in the manufacture of turkey-red oil. In all substitutes made by Henriques these sulpho-oleic acids were formed, in greater or less quantity, with a consequent diminution of substituted fatty acids. These concomitant reactions are difficult to regulate, for even when working under apparently identical conditions, he obtained variable proportions of insoluble fatty acids. As saponification eliminates chlorine from the molecule of the substitute, it was to be foreseen that the isolated acids would appreciably absorb more iodine than the substitutes from which they were derived, whilst the ordinary fatty acids yield, as is known, an iodine value approaching that of the oils from which they were derived. In fact, *the iodine values of the fatty acids are almost triple those of the substitutes.* Little is known of the action of chloride of sulphur (S_2Cl_2) on fatty oils. Warren asserts that drying oils yield solid masses with chloride of sulphur, *insoluble* in carbon disulphide, whilst the non-drying oils yield products *soluble* in that solvent. Stolmann, in the last

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(German) edition of Muspratt's *Dictionary*, writes that these results merit but little reliance, because it is known that olive oil—the type of the non-drying oils—is transformed by the action of chloride of sulphur into a mass analogous with rubber, insoluble in ether. The facts pointed out in Sommer's patent, as well as Henriques' results, formally contradict Warren's assertions.

If a sufficient quantity of chloride of sulphur be added to a fatty oil, the two liquids soon mix. After a few moments of contact energetic reaction sets in, accompanied by considerable disengagement of heat. The mixture froths, rises, gives off vapours of chloride of sulphur, with a little hydrochloric acid, sulphurous gas, and after a few seconds becomes converted into a solid, elastic, scarcely tacky mass, capable of being ground and crushed under the pestle. Exposed to the air, the mass loses the excess of chloride of sulphur employed and the adherent hydrochloric acid; it then resembles in every respect the white rubber substitutes of commerce. If one or other of the reagents, or both, be diluted with a neutral solvent, carbon disulphide or benzol, the reaction is longer in being manifested, its violence is moderated, but the final result is the same. The substitute is a little more porous in consequence of the volatilisation of the solvent. That is how the reaction goes on in presence of a sufficient quantity of chloride of sulphur. In the contrary case, along with a less disengagement of heat, a pasty, tacky residue is obtained, which even a long time afterwards, whether in the hot or in the cold, does not solidify. The quantity of chloride of sulphur required to transform an oil into a solid substitute varies with the nature of the oil, according to Henriques' experiments.

100 parts of oil of	do not yield a solid product with	Parts S_2Cl_2.	but do so well with	Parts S_2Cl_2.
Linseed		25		30
Poppy		30		35
Rape		20		25
Cotton		40		45
Olive		20		25
Castor		18		20

It will be seen from the inspection of these figures that there is no relation between the drying properties of oils and their aptitude to solidify under the action of chloride of sulphur. Having thus fixed the required proportions of chloride of sulphur, Henriques prepared and analysed substitutes with a linseed oil, rape oil and a poppy oil base, and with a mixture of equal parts of linseed oil and rape oil. The results are given in Table II., p. 79. Neither of these substitutes are analogous with the substitutes A and B of Table I., products of English origin, of which it would be desirable to know the method of preparation. On the contrary, the commercial sample C (Table II.) yielded, on analysis, figures so similar to those of the rape oil substitutes C, of Table I., that they may be rightly regarded as identical. The iodine values of the fatty acids isolated from substitutes present such wide differences from one kind to another that agreement in the iodine values may be taken as proof of identity. Moreover,

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Henriques *learned from a manufacturer that the bulk of the substitutes made in Germany are made from rape oil.*

The distinctive characteristic of the English substitutes A and B is their relatively low percentage of sulphur, according to which only 20 per cent. of chloride of sulphur had been used to solidify the oil. With the exception of castor oil, only the *oxidised* oils can be solidified with that proportion of chloride of sulphur.

Raw linseed oil, for example, which requires at least 30 parts of sulphur chloride, S_2Cl_2, to solidify it when it is fresh, only requires 15 to 18 per cent. when it has been heated for some hours at 200° to 250° C. (say 392° to 482° F.). If the temperature be pushed to 250° to 300° C. (482° to 572° F.), 10 per cent. of S_2Cl_2 suffices. A substitute prepared in that way would run into 4·78 per cent. of sulphur and 4·85 per cent. of chlorine. All drying oils behave in this respect like linseed oil.

By pursuing this method, Henriques identified the English substitutes with the product obtained by the action of chloride of sulphur on oxidised cotton-seed oil, known in the English trade under the name of soluble castor oil, "lardine".

Brown Substitutes.—These are met with in commerce sometimes in the form of deep brown, tacky blocks, sometimes in powder. Analysis shows the presence of a much greater quantity of sulphur than in the substitutes which we have previously examined. But chlorine is almost entirely absent. These substitutes are undoubtedly obtained by heating oil with sulphur. *They also dissolve in alcoholic soda;* the soap, treated by an acid, disengages appreciable quantities of *sulphuretted hydrogen;* the isolated fatty acids, however, contain a smaller proportion of sulphur than the substitutes from which they were derived. The iodine values of these substitutes and those of the fatty acids are rather high, from which Henriques concludes that it is linseed oil, or a mixture of linseed and rape oil, which is used in their manufacture. Henriques did not pursue the examination of these substitutes, which are much less interesting, from the scientific point of view, than those of which we have previously spoken.

It was interesting to ascertain whether the vulcanisation of rubbers, sophisticated with substitutes, influenced the percentage of chlorine in the product. Henriques examined a number of manufactured rubbers containing substitutes, and always detected chlorine in appreciable quantity. As no other chlorinated compounds are employed in the manufacture of rubber—with one exception, of which we shall speak later on—we may conclude that *the presence of chlorine in the alkaline alcoholic extract of a rubber is due to the use of a white substitute.* Quantitative tests, however, showed that the proportion of chlorine in manufactured rubbers is much less than that which corresponds with the quantity of substitute added. Thus, in two samples the percentage of substitute of which came out at 53 and 12 per cent., Henriques only found 0·5 and 0·37 of chlorine, whilst calculating on an average of 7 per cent. chlorine in the sub-

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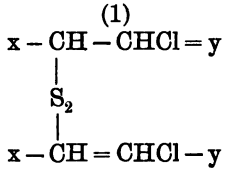
stitute, Henriques ought to have found 3·7 and 0·9 per cent. Cl. On vulcanisation, a portion of the chlorine is therefore disengaged either under the form of chloride of sulphur or as hydrochloric acid, or in some other way.

If alcoholic soda extracts a notable quantity of substance from a sample of rubber, and the extract contains no chlorine, we have the means of deciding whether the rubber is mixed with brown substitute or contains a fatty body. We have seen that the fatty acids liberated from substitutes contain a rather smaller proportion of sulphur than the substitutes themselves. Those of brown substitutes generally contain more than 10 per cent. If, therefore, the fatty oils have not absorbed sulphur during vulcanisation—if they have not by the heating itself of the rubber been transformed into substitute—we should be able, by isolating the fatty acids from the treatment with alcoholic soda, and by estimating their percentage of sulphur, to distinguish between the addition of an oil heated with sulphur and an ordinary oil. In order to solve this point experimentally, Henriques heated rape oil with an excess of sulphur for several hours at a temperature of 130° to 135° C. (266° to 275° F.), the highest temperature reached in vulcanisation. Under these conditions, the oil dissolved large quantities of sulphur, which for the greater part recrystallised out on cooling. After filtration the limpid oil was saponified, and the fatty acids separated in the usual way. Finally, the latter were dissolved in 90 per cent. alcohol to separate the precipitated sulphur, and the sulphur was estimated in the acids thus purified, in which were still deposited some crystals of sulphur after filtration. Found sulphur = 0·98 per cent.

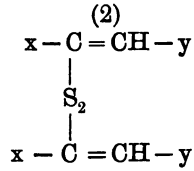
It will be seen that the quantity of sulphur found under these conditions may be neglected when compared with that which brown substitutes contain. The problem to detect in a rubber the presence of white substitute, brown substitute, or an ordinary fatty oil, and to estimate them therein, may therefore be regarded as solved. The presence of notable proportions of chlorine points to the addition of white substitute. The estimation of the sulphur in the fatty acids liberated from the alcoholic soda extract decides between brown substitute and a fatty oil. The method is at fault if a rubber contains all these three categories of substance simultaneously, but evidently that occurs very rarely.

Chemical constitution of white substitute (1) before, (2) after saponification :—

(1)



(2)



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CHAPTER VI.

THE MANUFACTURE OF DRIERS.

THE principal substances used as "driers" or aids in the process of oil-boiling are metallic lead (p. 33), oxides and salts of (1) lead and (2) manganese.

Lead Compounds Used as Driers.[1]—(1) Litharge; (2) red lead; (3) acetate or sugar of lead; (4) lead borate; (5) lead rosinate; (6) lead linoleate.

1. *Litharge*, PbO = 223; Pb = 92·83; O = 7·17 per cent. *Manufacture.*—When metallic lead is heated in a current of air, it is oxidised into massicot, PbO. 207 parts of lead yield 223 of massicot. When this massicot is further heated, it fuses, and is known as litharge, or, from its scaly nature, as *flake* litharge. It does not, however, undergo any chemical change. When buying litharge for use as a drier, it should be guaranteed free from copper, which is often present in considerable quantity, and acts injuriously. Moreover it should not contain unconverted metallic lead.

Properties.—Litharge should dissolve completely in dilute nitric acid and in acetic acid to a colourless solution. A green coloration would indicate copper. Any residue will consist, most probably, of grit; acetic acid may leave a residue of non-oxidised blue lead. When litharge is heated with linseed oil for some time at the temperature at which that oil is said to "boil," but, more properly, at which it undergoes destructive distillation, it dissolves in the oil. In varnish-making and oil-boiling it is often found at the close of the operation reduced to metallic lead. In such cases very probably all its oxygen is seized hold of and absorbed by the "boiling" oil. Litharge assists in oil-boiling in three different ways: (1) It may combine with any free linoleic acid in the oil at a comparatively low temperature to form linoleate of lead, which will dissolve in the oil as soon as it forms; (2) at a higher temperature it will eliminate glycerine from the oil and combine with the liberated linoleic acid to form linoleate of lead, which will again dissolve in the oil as soon as formed; (3) in the act of being reduced to metallic lead, it will part with its oxygen to the oil, and consequently by starting the oxidation process increase the drying properties of the oil. The drawback attendant on the use of litharge and all lead salts as driers is the dark colour

[1] Galen (131-200 A.D.) states that "white lead and litharge thicken and dry". —*De Meth. Med.*, iii., 4.

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which they impart to boiled oil, due no doubt to the dissolved lead linoleate. This dark colour is intensified by the great heat at which the oil must be kept for some considerable time before the litharge dissolves and plays its part. Another drawback attendant on the use of litharge and other lead salts is that the oil or varnish into which they enter cannot be used in conjunction with or in juxtaposition to pigments consisting of or containing sulphides liable to combine with lead compounds to form the black sulphide of lead. Sulphur emanations act similarly. Paintings or decorations injured in this way may, it is said, be revivified by washing with a weak solution of peroxide of hydrogen, which converts the black sulphide of lead into white sulphate of lead without materially affecting the painting in any other way. But this is erroneous. Thenard only used it to restore a white lead background in a black crayon drawing of Raphael. Such colours as madder lake would be destroyed by hydrogen peroxide. This black coloration is very often due to the free sulphur contained in the pigment, and not to the combined sulphur, which, looked at from a rational point of view, must be regarded as more or less stable. But, on the other hand, linseed oil boiled over the naked fire with litharge gives an elastic, durable coat of greater lustre and less susceptible to dry superficially than manganese boiled oil. In a word, litharge boiled oil is more of the nature of a varnish than manganese boiled oil.

2. *Red Lead Minium*, Pb_3O_4 = 685; Pb = 90·2; O = 9·8 per cent. *Manufacture.*—Red lead Pb_3O_4 is made by heating litharge in contact with air at 300° C., a temperature slightly below its point of fusion. The litharge should be free from copper. It gradually becomes converted into a fine red known as red lead. The manufacture of red lead is a Derbyshire industry, and the process of the present day differs in no essential particulars from that described so vividly by Bishop Watson 125 years ago. A variety of red lead, made by igniting white lead, is sold as orange lead. Although dearer in price it is no more efficacious as a drier than ordinary red lead. It always contains undecomposed carbonate.

Composition.—The red leads of commerce differ in composition according to the length of time they have been furnaced. However, the proportion of oxygen absorbed by the litharge never exceeds that which corresponds to the formula $2PbO,PbO_2 = Pb_3O_4$. When red lead is heated it darkens, and then, at a temperature above 300° C., it gives off oxygen, becoming reconverted into litharge. *Test for Purity.*—When red lead is treated with nitric acid the two equivalents of PbO are dissolved as nitrate of lead, leaving the PbO_2 behind as a puce-coloured residue which is perfectly insoluble in nitric acid, and only dissolves in dilute nitric acid after it has been reduced by the addition of oxalic acid. When this reagent is introduced into the test tube, containing the nitric acid and red lead, vigorous action ensues, the whole of the puce-coloured oxide being converted into white crystalline nitrate, to which it is only necessary

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to add a little boiling water for complete and instantaneous solution. Any residue remaining is generally barytes. Sophistication with ground brick is only met with in books. Red lead, owing to its containing more oxygen perhaps in a more available state, may be regarded as a more energetic drier than litharge, and its function as a drier may be regarded as similar to what it plays in a storage battery.

Other Uses.—Besides its use as a drier, red lead is used in the manufacture of wall paper, sealing wax, crystal glass; mixed with white lead in oil it is used to form steam joints. Red lead "substitutes" are of little or no use as driers.

3. *Lead Acetate* (*Neutral*), $Pb(C_2H_3O_2)_2 + 3H_2O$. *Sugar of Lead. Molecular Weight*, 379.—This salt was formerly known as *salt of Saturn*, or sugar of Saturn. It is prepared by neutralising acetic acid with litharge. It crystallises in oblique rhomboidal prisms, colourless, transparent and efflorescent. It is generally met with in the form of heavy compact crystalline masses, somewhat resembling loaf-sugar. Its odour is acetous, whilst its taste is both sweet and astringent, its after taste is bitter and disagreeable. It dissolves in $\frac{1}{2}$ part of cold water and in 8 parts of alcohol. Its solution slightly reddens blue litmus paper, and is partially decomposed by carbonic acid, which at the same time liberates a small quantity of acetic acid, which preserves the rest of the salt from the action of carbonic acid.

Action of Heat.—Crystallised acetate of lead melts at 75·5° C., at 100° C. it loses water and a small quantity of acetic acid. It afterwards solidifies, but towards 280° C. it again melts. The dehydrated salt would appear to consist of a sesquibasic acetate. At higher temperatures it decomposes with disengagement of carbonic acid, acetic acid and acetone, leaving a residue of very finely divided and highly combustible metallic lead. Lead acetate is a very energetic drier; perhaps much of the metallic lead found by varnish-makers at the bottom of their pots may come from the complete decomposition of the acetate and not from the litharge often used in conjunction with it.

3A. *Basic Lead Acetate.*—The so-called neutral lead acetate just described results from the combination of two equivalents, *i.e.*, 120 parts of monobasic acetic acid with one equivalent, *i.e.*, 223 parts of the di-acid base litharge. When *two* equivalents of a *monobasic* acid combine with *one* equivalent of a *di-acid* base a neutral or normal salt is produced. But it is found in actual practice that certain neutral or normal salts, in their state of solution in water, can still dissolve and combine with a further proportion of base. Thus when one equivalent of neutral acetate of lead is dissolved in water, it combines on boiling with an additional equivalent of litharge to form the di-basic salt, $PbA_2Pb(HO)_2$. By digesting together one equivalent of sugar of lead with two equivalents of litharge the tribasic salt, $Pb(A)_2 2Pb(HO)_2$, is produced. These basic acetates, owing to the fact that their excess of litharge, or, rather, lead hydrate PbO,H_2O,

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is in a loose state of combination, easily give up this excess to weak acids, even carbonic acid, as exemplified in the manufacture of white lead. Should a raw linseed oil contain free linoleic acid, agitation with a solution of basic acetate of lead will convert the former into linoleate of lead, and if the agitation be renewed from time to time this linoleate of lead will dissolve in the oil imparting drying properties thereto, whilst the insoluble magma produced by the action of the basic acetate on the colouring matter and mucilage collects as an insoluble slime at the bottom of the vessel. Such is the principle of the refining of linseed oil by Liebig's method and the imparting of drying properties thereto in the cold by means of basic acetate of lead, a substance the utility of which is not appreciated either by the varnish-maker or colour-maker to the extent to which its intrinsic merits entitle it.

4. *Borate of Lead,* $Pb(BO_3)_2 + H_2O = 310{\cdot}8$.—Borate of lead is a white substance made by precipitating the boracic acid in 382 parts (*i.e.*, one equivalent) of borax ($Na_2B_4O_7 + 10$ aq.) by one equivalent of a lead salt—that is to say, 331 parts of nitrate of lead $Pb(NO_3)_2$ or 379 parts of neutral acetate of lead ($Pb(C_2H_3O)_2 + 3$ aq.). The mother liquor is filtered off, the precipitate is well washed, filter-pressed and dried in the usual way. This substance combines the drying properties of both lead and boracic acid. It does not darken the oil to quite the same extent as other lead driers.

5. *Rosinate of Lead.*—The first stage in the manufacture of rosinate of lead, erroneously termed resinate of lead, is the preparation of an alkaline solution of rosinate of soda. Rosin being an acid body of variable degrees of acidity, different samples require different proportions of caustic alkali for exact neutralisation. In actual practice the best plan is to add rosin gradually and with constant stirring to a very dilute boiling solution of caustic alkali of known strength until the latter is exactly neutralised. 100 lb. of rosin take about 22 lb. of 77 per cent. caustic.

Starting from the data that every 62 parts of anhydrous sodium oxide (real alkali) present in solution as rosinate of soda require for complete precipitation one equivalent of a lead salt, *viz.*, 379 parts lead acetate or 330 parts lead nitrate for complete precipitation, and knowing the original bulk of the alkaline solution used and its richness in real alkali, as determined by the hydrometer and the usual tables, it is easy to calculate the amount of lead salt necessary to displace the soda in the solution of alkaline rosinate by lead, and thus throw down all the rosin as rosinate of lead. (See Saponification Value, pp. 114-117.)

The dilute solution of the proper proportion of lead salt is gradually added with constant stirring to the equally dilute solution of alkaline rosinate; the mother liquor is syphoned off, the precipitated lead rosinate, which is further well washed, drained and dried. It may be heated to incipient fusion to expel water.

6. *Linoleate of Lead.*—Linoleate of lead is made by precipitating a solution of linseed oil soft soap, made by saponifying linseed oil with

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caustic potash in the usual way. 100 lb. of linseed oil on an average require 19½ lb. of pure caustic potash, equal to 16½ lb. of anhydrous potassic oxide, for complete saponification. (See Sap. Value, pp. 114-117.) Caustic potash is sold as containing so much per cent. of anhydrous caustic alkali; when pure it contains 83·93 per cent. It is, therefore, a mere matter of calculation if 100 lb. of linseed oil require 19½ lb. of caustic potash, when the strength is 83·93 per cent., to find how much will be required for complete saponification when the strength is 80 (or any other figure) per cent. The soap is dissolved in five or six times its weight of water, and the linoleic acid precipitated therefrom by a dilute solution of a lead salt. The exact proportion of lead salt for complete precipitation depends upon the amount and the strength of the caustic alkali used. 112·2 parts of pure caustic potash, equal to 94·2 parts of anhydrous potassic oxide, require one equivalent of a lead salt, say 379 parts lead acetate or 330 parts lead nitrate. The lead salt solution is added gradually to the soft soap solution with constant stirring. The mother liquor is syphoned off the precipitated lead linoleate, which is then further washed, drained and dried. Heat after drying to incipient fusion to expel last traces of water.

Manganese Driers.—The manganese compounds used as driers are: (1) Black oxide—MnO_2; (2) manganese sulphate—$MnSO_4$; (3) manganese acetate—$Mn(C_2H_3O_2)_2$; (4) manganese borate; (5) manganese oxalate; (6) manganese linoleate; (7) manganese rosinate. Metallic manganese is a reddish-grey coloured brittle metal, difficultly obtained from its naturally occurring oxides by heating strongly with charcoal. Finely divided metallic manganese has such affinity for oxygen that it decomposes water at the ordinary temperature, with evolution of hydrogen gas. In order to preserve it in an unoxidised condition it must be preserved in a hermetically sealed tube or under naphtha. This strong affinity of metallic manganese, as well as its compounds, for oxygen constitutes in great part the value of manganese ores for commercial purposes. This value is further greatly enhanced by the fact that the oxidised compounds of manganese, under certain circumstances, easily part with a portion of their oxygen, becoming converted into lower oxides, and these lower oxides, in contact with the air, reabsorb oxygen, and are reconverted into the original higher oxides. The drying properties which manganese oxides and salts impart to oils boiled in contact with them was said to be due to the fact of these oxides and salts being so easily deoxidised to a certain extent, and then just as easily reoxidised—*e.g.*,

$$3MnO_2 = Mn_3O_4 + O_2, \text{ and again}$$
$$Mn_3O_4 + O_2 = 3MnO_2.$$

But, as a matter of fact, linseed oil is not oxidised to any very great extent in oil-boiling.

1. *Black Oxide of Manganese Pyrolusite*, $MnO_2 = 87$; $Mn = 63·22$; $O = 36·78$ per cent.—This is the source from which the various salts of manganese are prepared. Not only is it the most im-

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portant, but also the most widely distributed of manganese minerals. It usually occurs in heavy compact masses, reniform, and sometimes with a fibrous or radiated structure. The colour is iron black to dark steel grey, and very often soils the fingers. It gives with unglazed porcelain a grey streak. The other manganese minerals give a brown streak. When heated alone it liberates considerable quantities of oxygen, and becomes converted into trimanganic tetroxide, Mn_3O_4. Pure pyrolusite has a specific gravity of 4·9. In commerce pyrolusite is usually found as a coarse, dull, black powder, consisting of a mixture of manganese dioxide, sesquioxide and monoxide, and contaminated with the gangue, quartz, felspar, limestone, etc., which often amount to 40 or 50 per cent. Besides being used directly as a drier, pyrolusite—*savon des verriers*—is extensively used in glass manufacture to burn up any carbonaceous matter and at the same time impart a slight violet tint, which masks the usual yellow tint of glass.

2. *Sulphate of Manganese,* $MnSO_4 5H_2O = 151 + 90$ aq.—This beautiful rose-coloured salt is prepared on the large scale by heating pyrolusite with coal and dissolving the impure monoxide, to which it has been reduced, in sulphuric acid. A little muriatic acid is added towards the end of the operation. The solution thus obtained is evaporated to dryness and afterwards exposed to a red heat, so as to decompose any sulphate of iron. The pure sulphate is filtered and drained from the insoluble oxide of iron. It crystallises with: $7H_2O$ below 6° C.; $6H_2O$ between 7° and 20° C.; 3 to $4H_2O$ at higher temperatures; $1H_2O$ at 200° C. The monohydrated salt $MnSO_4 + H_2O = 169$ is greyish-white in colour, and is the starting-point of the manufacture of all manganese driers.

3. *Manganese Acetate.*—This salt is manufactured by double decomposition between acetate of lime and sulphate of manganese in equivalent proportions.

The reaction is as follows:—

$Ca(\bar{A})_2 +$	$MnSO_4 =$	$CaSO_4 +$	$Mn(\bar{A})_2.$
Calcium acetate	Manganese sulphate	Calcium sulphate	Manganese acetate.

The brown acetate of lime may be used. A solution of acetate of manganese marking 22° Twaddell, specific gravity 1·110, is used in oil-boiling by steam in the proportion of 36 gallons to 10 tons of oil in conjunction with a small quantity of lead driers, the greater the proportion of which the darker is the resultant boiled oil. (See Vincent's process of oil-boiling by steam (p. 49), for which process this drier is especially applicable.)

4. *Manganese Borate.*—This is prepared by precipitating a solution of 382 parts, *i.e.*, one equivalent of borax, $Na_2B_4O_7 10$ aq., with 169 parts, *i.e.*, one equivalent of the monohydrated sulphate of manganese, $MnSO_4,H_2O$. The sulphate of manganese should be free from iron and the borax from excess of alkali. The iron may be removed by roasting the sulphate and redissolving. The excess of alkali may be

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neutralised by the addition of the requisite quantity of boracic acid. Precipitation should be effected in the cold. This substance, when pure and skilfully prepared, is a perfectly white powder, but whiteness is not a guarantee of purity, as it is often most grossly sophisticated with sulphate of lime, zinc oxide, etc.

It is sometimes very unskilfully prepared. Some commercial samples submitted to J. G. McIntosh for analysis consisted of mere mechanical mixtures of sulphate of manganese and borax almost entirely destitute of drying power.

Endemann and Paisley found the following percentages in four samples:—

1.	Boric acid,	39·36;	manganous oxide,	12·00.
2.	,,	,,	40·18;	,,	,,	16·19.
3.	,,	,,	37·38;	,,	,,	31·06.
4.	,,	,,	11·09;	,,	,,	6·43.

This salt is also made by acting on borax with manganous chloride and is supposed to be represented by the formula MnB_4O_7, but the washing with water to get rid of the sodium chloride also formed partly decomposes it, and the salt is very apt to turn brown from oxidation. Many manufacturers use a large excess of borax, and simply drain the precipitate without washing it. In the above analyses No. 3 was a washed product which had been bleached with sulphate of sodium. No. 4 contained rosin.

When manganous sulphate is used in the manufacture of borate of manganese the yield is less than with the chloride, as the sodium sulphate dissolves quantities of the manganese borate.

By reacting on manganous sulphate with the equivalent quantity of borax (see p. 89), Endemann and Paisley obtained precipitates which, in two cases, contained 26·78 and 28·82 per cent. of manganous oxide and 42·36 and 39·69 per cent. of boric oxide (B_2O_3). If the salt were MnB_4O_7 the amounts of B_2O_3 for these two percentages of manganese should be 52·8 and 56·8 per cent. respectively. Endemann and Paisley recommend the following process for the manufacture of borate of manganese: Manganous chloride is precipitated with borax mixed with enough caustic soda to double the amount of soda present. The precipitate is turbined, washed twice with a little water. The loss of boric acid is found by analysing the wash water, and is replaced by mixing solid boric acid with the nearly dry precipitate, and drying the mixture thoroughly. It may then be regarded as MnB_4O_7, with either three or five molecules of water. Borate of manganese prepared in this way gave capital results as a drier.

5. *Manganese Oxalate.*—Manganese oxalate is made by precipitating a solution of oxalate of soda by a manganese salt. In boiling the oxalic acid of the manganese oxalate is decomposed with evolution of CO_2, etc. Very energetic oxidising properties are claimed for the residual manganese.

6. *Manganese Linoleate.*—Manganese linoleate is made in the same way as lead linoleate. A linseed oil soft soap is precipitated by

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a solution of the monohydrated sulphate of manganese, 169 parts of which, or one equivalent, are equal to 112·2 parts, or two equivalents of pure caustic potash which contain 83·3 parts of anhydrous potassic oxide (the percentage of real alkali contained in pure caustic potash), and as 100 parts of linseed oil require 19½ parts of pure caustic potash for complete saponification, it is easy to make the necessary calculation when working with potash of any strength different from the pure, *viz.*, 83·3 per cent. of real alkali. Or potash lye of a certain gravity may be kept in stock, and its richness in real alkali ascertained from the customary tables and the gravity as indicated by the hydrometer. For 100 lb. of linseed oil caustic potash equal to 16½ lb. of real alkali must be used, starting initially with a weak solution and finishing with a more concentrated. The soft soap of commerce contains about 50 per cent. of water, and though often made from linseed oil is not invariably so, and, moreover, it is grossly adulterated with starch, silicate of soda, and so on. The varnish-maker who makes his own driers should, therefore, make also his own soft soap.

Manganese linoleate is precipitated, washed, pressed and dried in the same way as lead linoleate.

7. *Rosinate of Manganese.*—Rosinate of manganese is made in the same way as lead rosinate; for every 80 parts of pure caustic soda (containing 62 parts of pure anhydrous alkali sodium oxide) present in the solution of rosinate of soda, 169 parts of monohydrated sulphate of manganese are required for precipitation.

Mixed rosinates of manganese and lead and mixed linoleates of manganese and lead, as well as mixed rosinates and linoleates of both lead and manganese, can easily be made in the wet way, *e.g.:* Saponify equal weights of rosin and linseed oil and precipitate by a solution of a mixture of the acetates or nitrates of manganese and lead in molecular proportions. Product in turps is a fine drier.

Fused rosinates and linoleates are made by dissolving the oxides of lead and manganese in rosin or in linseed oil respectively. The proportions may be calculated as follows: as the atomic weight of KHO 56·1 is to the saponification value of the rosin or linseed oil, say, 17·5 and 19·5 per cent. respectively, so is half the atomic weight of litharge 111·5 and manganese dioxide 43·5 to the amount of litharge or manganese required per cent. Commercial articles are often grossly adulterated.

Resinates of Manganese and Lead.

The true resinates of manganese and lead are made by saponifying such resins as copal by caustic soda and reprecipitating by a salt of lead or manganese. They form interesting compounds, but they are even more intractable as far as solution in oil is concerned than the original resins from which they are derived.

Patent Driers: Concentrated Driers and Liquid Driers.—Concentrated driers consist of the product obtained by heating linseed

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oil at 250° to 300° C. to the consistency of sticking plaster, with a quantity of litharge, red lead, borate of manganese greater than 10 per cent. and even as high as 70 per cent. of the weight of the oil. Sugar of lead, oxide of zinc, etc., are added to or partially substituted for the preceding substances.

Concentrated driers consist of a thick viscous mass, generally of a deep brown colour, composed essentially of fused linoleate of lead. A good product may be made by boiling in a small pot 7 parts (by weight) of good and aged linseed oil—old tanked oil—with a mixture of 2 parts of litharge in powder and 2 parts of red lead. The oil at first assumes a beautiful red colour, which turns brownish as the temperature rises. Suddenly it thickens, looking like bronze in fusion, whilst at the same time abundant fumes are given off. A sample drop spotted on a glass plate should become in a minute perfectly solid, with no viscosity nor tackiness whatever. It is thinned down with spirits of turpentine or rectified naphtha to the requisite consistency when required for use. Concentrated driers dissolved in benzol or spirits of turpentine are used to make "boiled" oil or oil "boiled through the bung-hole" in the cold from raw linseed oil.

Terebine or liquid drier is made like concentrated driers, but when the pot is taken off the fire it is only allowed to cool for a few minutes, when it is thinned down with successive additions of spirits of turpentine, with constant stirring. It is then passed through coarse linen, and stored in closed vessels. Liquid driers consist therefore of linseed oil saponified and oxidised to a greater or less extent, *i.e.*, heated until it will roll into pills, and afterwards dissolved in spirits of turpentine. There are numerous recipes for making driers; *inter alia* the following:—

Concentrated Driers.

7 lb. linseed oil.	7 lb. linseed oil.	7 lb. linseed oil.
2 lb. litharge.	2 lb. sugar of lead.	1 lb. umber.
2 lb. black oxide of manganese.	2 lb. red lead.	1 lb. litharge.
1 lb. red lead.	2 gallons spirits of turpentine.	1 lb. oxide of manganese.
2 gallons spirits of turpentine.		1 lb. sugar of lead.
		2 gallons spirits of turpentine.

Pale Liquid Driers—Terebines.

7 lb. linseed oil.	7 lb. linseed oil.	7 lb. linseed oil.
3 lb. pure dry white lead.	2 lb. borate of manganese.	2 lb. borate of manganese.
1½ lb. sugar of lead.	2 lb. zinc white.	1½ lb. acetate of lead.
2 gallons spirits of turpentine.	2 gallons spirits of turpentine.	2 gallons spirits of turpentine.

In the manufacture of white liquid driers the mass does not become red on boiling, but white at first, and afterwards a very faint yellow. Liquid driers are but little used for white colours —solid driers are preferred; but with other coloured paints very rapid drying is effected by simply adding, say, a few parts in 100

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of liquid driers to the raw oil used. Here we have a case of oxidation analogous to that pointed out by Chevreul, *viz.*, when a manganese-boiled oil is added to raw oil. Endeavours have been made by working in the cold to produce very pale liquid driers. One variety which is used very extensively is made by intimately mixing 100 parts (by weight) of finely pulverised sugar of lead with 1,200 parts (by weight) of poppy-seed oil. This mixture is exposed to sunlight in a glass vessel, and frequently stirred. There is got in this way a perfectly colourless oil, which, when thinned down with 250 parts (by weight) of spirits of turpentine, dries very rapidly, yielding a solid, durable coating. *Hartley and Blenkinsop's drier* consists of zinc oxide mixed with a solution of linoleate of manganese dissolved in naphtha. One per cent. of manganese linoleate added to linseed oil in a dilute naphtha solution renders it quick drying. There is no necessity to blow air through it. The air will do its work very well after the oil is applied to any given surface.

The so-called " terebine " very often consists simply of japanners' gold size thinned down with spirits of turpentine. Japanners' gold size is linseed oil boiled with litharge, red lead, sugar of lead and zinc sulphate until it will roll into pills, and then dissolved in spirits of turpentine. Another process consists in fusing 2 parts of Manilla copal, which is then mixed with ½ part of linseed oil and 6 parts of concentrated driers made by one of the formulæ given above, after which the whole is incorporated with 14 parts of spirits of turpentine. The bulk of the *paste "patent driers"* used in the trade consist of 90 per cent. or more of a mixture consisting of equal quantities of chalk and barytes and about 10 per cent. or less of actual driers. When it is desired to prevent the paint from cracking, very good results are obtained by using spirits of turpentine which has simply been shaken up with litharge and decanted. A liquid is thus obtained which does not affect the colour, and which also gives a very durable coating.

Hannay's glyce lead linoleates and basic lead linoleates, made by dissolving about 2 parts (by weight) of litharge in 1 part of linseed oil at as low a temperature as practicable, should make excellent driers either *per se* or in turps or benzene.

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CHAPTER VII.

THE DETECTION OF ADULTERATION IN LINSEED AND OTHER DRYING OILS BY CHEMICAL, PHYSICAL AND ORGANOLEPTIC METHODS.

1. *Smell.*—It is almost impossible to add any oil to linseed oil which has got a pronounced odour without that odour being almost as pronounced in the raw oil as it is in the original adulterant. Rosin oil and fish oils make themselves felt in linseed oil by their characteristic odour. Fish oil is perhaps not so readily revealed by its smell in boiled oil, but it manifests itself even then when energetically rubbed between the palms of the hands, as if one were making a lather, and then applied to the nostrils. The characteristic smell of burning rosin is developed when the oil is burned, if it should contain either rosin or rosin oil. The smell of the pure genuine oil is very characteristic, especially when newly pressed; it then has a bland, mucilaginous, appetising odour, but this odour gives place as the oil ages to a somewhat rank, but not unpleasant, though still characteristic smell. The newly pressed oil should not give off the smell of cooked turnips which have been overheated in the operation. That is a sign that the oil has been made from seed containing rape, or other cruciferous oils, and that it has been extracted at too great a steam heat. The nose is a very useful factor in the testing of oils, and no opportunity should be lost in training it and exercising it in its duties in this respect. But one must not be led away even by a smell into the fields of imagination; one must not imagine himself in Dante's Inferno every time one smells sulphur.

2. *Taste.*—The taste of linseed oil is also as characteristic as its smell; when new it is bland and pleasant; old oil is slightly acrid. Fish oils can hardly be detected in linseed oil by their taste unless present in large proportion, but the most minute trace of rosin oil can most easily be detected. When a linseed oil containing rosin oil first impinges on the palate nothing very much amiss is discovered at first, and the operator thinks, as far as taste is concerned, the oil is all right, and perhaps if uninitiated goes away satisfied to perform some other test on the oil, but by-and-by a bitter, biting, acrid, nauseous taste makes itself felt on the back part of the palate, which, in pronounced cases, makes one shudder and expectorate to get quit of it. But it remains persistent for some considerable time. There

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is no necessity to recall the taste of rosin oil, one remembers it all one's life.

An abnormal uncommon smell in linseed oil is generally due, fish oil and rosin oil excepted, to some volatile oil or another. When water is distilled over linseed oil, or when linseed oil is treated with a current of steam, it should yield no other distillate than condensed water; a volatile distillate would point to spirits of turpentine or benzene.

3. *Colour of Raw Linseed Oil.*—The colour of *raw linseed oil* is usually a brownish-yellow; when refined it is almost colourless. A blue or green fluorescence exhibited when the oil is looked at on a black background with the back to the light is a sure sign of adulteration with either mineral or rosin oil. This bloom may be somewhat removed from the rosin oil by the addition of ½ lb. to 1 lb. of di-nitro-naphthalin to every 10 gallons of rosin oil, but the method is too costly; besides the fluorescence has a tendency to return. Moreover, the nitro-naphthalin renders the oil acid, and, besides, it throws the rosin or mineral oil several shades back in darkness of colour. (See vol. ii.)

The Colour of "Boiled" Linseed Oil.—This may vary from the pale colour of the raw oil itself to port wine or even porter colour, the latter colour being characteristic of oils from which the drier has not had time to settle out. Fluorescence or bloom should be carefully looked for by spotting a drop of the oil on black paper, or on the black cover of a note-book, and turning the back to the light, lowering the paper to the knees, and gradually raising and lowering it at several inclinations. If any rosin oil or mineral oil be present it generally shows itself by the bloom manifested under these circumstances. But the bloom is not so readily seen in boiled as in raw oil, especially when the rosin oil has been boiled along with the linseed. When the rosin oil is boiled along with the linseed, so long as not more than 2 or 3 per cent. of rosin oil is used, the practice is not quite so bad as at first sight might appear; the acidity of the rosin oil is corrected by the litharge, and the rosin acids are converted into rosinate of lead, and thus assist in stimulating the drying propensities of the oil, and the profit on this small addition pays for the cost of boiling. The painter does not care to pay much higher for his boiled than he does for his raw oil, and to this extent he shows himself unreasonable. When the oil is full of flaky matter, and even after filtration continues to deposit foots, it points to cotton-seed oil having been boiled with the oil. Its stearine separates out *ad infinitum.*

4. *Specific Gravity.*—The specific gravity of linseed oil at any given temperature is its weight compared with the weight of an equal bulk of water at the same temperature taken as unity. Sometimes, however, the density at 100° C. (212° F.) is referred to water at 15° C., or 60° F. (15·5° C.), and so on. The reason of this is that the specific gravity bottle which is used to take the density of the oil is graduated at, say, 60° F. Moreover, the difficulties in graduating a specific

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gravity bottle at 212° F. are sufficiently obvious. The specific gravity bottle may be had graduated to hold 1,000 grains at 60° F. or 50 grammes or 100 grammes. When the bottle and stopper are tared in the case of the 1,000 grain bottle, the number of grains added to restore equilibrium give directly the specific gravity of the oil, but in the case of the 50 grammes or 100 grammes the exact weight added to restore equilibrium has to be multiplied by 20 or by 10 respectively. For all practical purposes the density of linseed is taken with sufficient accuracy by the hydrometer. The most useful hydrometer for linseed-oil testing is graduated from ·900 to ·950, and costs about fifteen to eighteen pence, and a suitable thermometer may be had for half a crown. The oil-refiner and boiler could not invest in two more serviceable instruments. Life is too short for taking the specific gravity of linseed oil by specific gravity bottles or Westphal balances. It is only wanted to the third decimal point, and that is given correctly by any accurate hydrometer. It will be seen that no two observers agree in regard to the gravity of linseed oil at any given temperature. Possibly some of these variations are due to experimental errors, but as a matter of fact it would be very difficult to get two samples of oils representing different bulks to coincide exactly in density, and not only in density, but in other particulars, and it may be advisable to discuss here, once for all, the reasons for

FIG. 23.—Thermo-hydrometers for Oil.

DENSITY OF LINSEED OIL.[1]

Degrees C.	Density.	Observer.	Degrees C.	Density.	Observer.
12	0·939	Saussure	15·5	0 9316-0·9345	Lewkowitsch
15	0·932-0·937	Allen	17·5	0·9305-0·9370	Filsinger
15	0·9305-0·9352	Wijs	18	0·9299	Stillwell
15	0·9325	Souchère	18	0 9411	,,
15	0·9347	Schiebler	20	0·9275-0·935	Holde
15	0·9315-0·9345	Thomson and Ballantyne	25	0·930	Saussure
			50	0·921	,,
15	0·9342	De Negri and Fabris	94	0·881	,,
15·5	0·931-0·937	McIlheny	[2]99	0·8809	Allen

[1] Add or subtract 0·004 for every 10° F. above or below 60° F.
[2] Water at 15·5° C. = 1.
Density of fatty acids of linseed oil—
at 15·5° C. = 0·9233 (Allen).
at 99° C. (water at 15° C. = 1) = 0·8612 (Allen).
at 100° C. (water at 100° C. = 1) = 0·8925 (Archbutt).

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this continual variation. The density of linseed oil, like all its other so-called chemical and physical "constants" (*sic*), varies (1) with the general purity and with the pedigree of the seed from which the plant which produced it sprung; (2) with the soil on which the plant producing the seed (which when crushed yielded the oil) grew; (3) with the climate in which that soil was located; (4) with the manure applied to the crop; (5) with the time of sowing; (6) with the previous rotation of crops; (7) with the season whether wet or dry; (8) with the after cultivation; (9) with the time of harvesting, *i.e.*, whether the seed was allowed to ripen too little or too much, *i.e.*, whether or not the exact moment was seized for harvesting the crop, when the oil in the seed was in that state of maturity in which, after rational harvesting of the flax, threshing and storage, and winnowing of the seed and expression therefrom of the oil, the latter excels in all those good qualities for which linseed oil is so justly and so highly esteemed. Independently therefore of impure seed and adulteration of the oil, whether by intentionally crushing it with a greater or less amount of seed which is not linseed, or after expression by the addition of an oil previously expressed from seed which is not linseed, or by the addition of marine animal oils (fish oil, whale oil, etc.), mineral oils or rosin oil, the density of genuine linseed oil, like every other so-called chemical and physical constant, varies within rather wide limits. It is a very difficult matter indeed to convince the uninitiated that the properties of genuine linseed oil are not like the law of the Medes and Persians, "which altereth not". But having gone into the reasons why raw linseed oil varies in composition and properties, it will only be necessary to refer to the matter again when it is especially important to do so.

Presuming, however, that we are dealing with a genuine oil, the density should never go below 0·931 nor above 0·934 in the case of raw linseed oil. Books and trade journals give higher densities, but the oil is not to be met with on the market with a higher density than 0·934; even 0·933 is rare. The linseed-oil consumer is to a great extent indebted to the farmer for the purity of the oil supplied to him. The crusher must needs sell his oil-cake, and as, *e.g.*, rape seed can be detected under the microscope and also by chemical means in the linseed cake, the crusher cannot very well crush a certain amount of non-drying oil with his linseed should it yield an oil of very high gravity. He can, of course, sophisticate it afterwards, but some crushers imagine that if they crush the two seeds together, and thus treat the oil exactly alike, that the fraud will escape detection. So it may if the linseed oil from that particular batch of seed be of itself of high gravity and yield high tests generally. But then the crusher would be in hot water with the farmers and the agricultural societies to which they belong; his oil-cake would get a bad name, and in the end the crusher would only prove once more that honesty is the best policy.

Oils of Similar Gravity to Linseed Oil.—The only oils approaching

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linseed oil in gravity are Lallemantia oil and certain fish or marine animal oils, which can be detected by methods given in the sequel.

Oils of Different Gravity to Linseed Oil.—All other vegetable drying and non-drying oils are specifically lighter than linseed oil, with the exception of wood oil, castor oil, croton oil, rosin oil, the hydrocarbide oil produced by the destructive distillation of rosin. When linseed oil is dear there might be a tendency to adulterate it with lower grade castor oil. The greater density, unless manipulated to meet the case by the addition, say, of rape oil, would at once indicate the sophistication, but here comes in the bad effect of the high maximum densities given in books. Supposing 10 per cent. by volume of castor oil 0·960 gravity were added to a linseed oil of 0·931 gravity,

We get 9 × 0·931 = 8·379
„ „ 1 × 0·960 = ·960
10)9·339
0·9339

Now, as far as density is concerned, no one can go into a court of law —in face of what is given in books—and swear that the above sample is not genuine linseed oil. But happily every particle of the castor oil can be separated by alcohol and weighed. So the sophisticator has to bear in mind that his oil must not satisfy one test alone; it must satisfy at least a score of tests. Rosin oil can be separated out from linseed oil almost as easily as castor oil; when the soft soap made from a linseed oil containing rosin oil is dissolved in water and shaken up with ether the latter extracts all the rosin oil and floats to the top of the soap solution from which it is decanted, the ether distilled and the rosin oil weighed as described in the sequel (p. 112).

TABLE SHOWING MONTHLY INCREASE IN DENSITY IN OILS EXPOSED TO AIR IN UNCORKED BOTTLES FROM ONE MONTH TO SIX MONTHS, THE BOTTLES BEING SHAKEN EVERY MORNING (THOMSON AND BALLANTYNE).

Oil.	Density, Original.	Densities at end of each Month.					
		1	2	3	4	5	6
Olive - - -	0·9168	0·9187	0·9193	0·9208	0·9215	0·9227	0·9246
Castor - -	0·9679	0·9681	0·9691	0·9700	0·9700	0·9685	0·9683
Colza - - -	0·9168	0·9183	0·9172	0·9185	0·9184	0·9200	0·9207
Cotton-seed -	0·9225	0·9237	0·9241	0·9261	0·9278	0·9304	0·9320
Arachis - -	0·9209	0·9213	0·9221	0·9233	0·9239	0·9256	0·9267
Linseed - -	0·9325	0·9331	0·9336	0·9353	0·9359	0·9372	0·9385

5. *Solidification Point of Linseed Oil.*—The temperature at which linseed oil concretes into a solid fat is a useful indication. But,

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unfortunately, authorities differ here as in every other physical or chemical "constant". It is greatly to be feared that many of the determinations have not been made upon pure samples of linseed oil. It serves no good purpose to make elaborate preparations and precautions to secure accurate results with the samples tested if they be impure or even contain a notable proportion of fatty acid. Before taking the solidifying point intended to form a standard for future reference and comparison, the oil in question should be pure and the fatty acids eliminated by treatment with alcohol, and all trace of the latter got rid of by gentle heating. Linseed, walnut and hemp-seed oils would appear to have much the same freezing-point, *viz.*, – 27° C. (– 17° F.), whereas cotton-seed oil has a freezing-point, *viz.*, – 2° C., very nearly that of water. But it must not be taken for granted that the freezing-points of mixtures of two or more oils can be calculated from the freezing-points of their ingredient oils. This would be a great mistake, and there is abundant evidence to show that mixtures of oils behave in this respect as abnormally as similar mixtures in the inorganic world, and just as mixtures of the carbonates of potash and soda melt at a temperature much lower than either carbonate does alone, so also does a mixture of cotton-seed oil stearic acid and stearic acid from tallow melt at a temperature below that of either of these varieties of stearic acid. Again Jean was baffled in his attempt to determine the melting-point of oils like linseed by mixing them with a known percentage of palmitic acid of known melting-point, but possibly he might have succeeded better had he used ceresin of high melting-point.

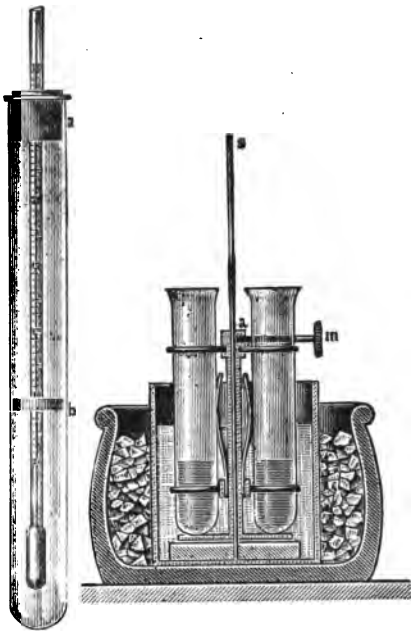

FIG. 24.—Apparatus for Determining Solidifying Point of Linseed Oil.

The melting-point of the fatty acids from linseed oil, *viz.*, 11° C., differentiates it from several other oils, especially cotton-seed oil, whose fatty acids solidify at + 28·5° to 30° C. But here, again, the utility of such data is negatived by the fact that, judging from analogy, the fatty acids from a mixture of the two oils in equal proportions would not be at all likely to yield a product with a freezing-point which would be the arithmetical mean of those of its constituents. Much information may be intuitively gained in regard to the solidification

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and melting-point of oils by observing the behaviour of the stock bottles and samples, filed away for reference, when a frost begins to set in, and again when the corresponding thaw ensues. Cotton-seed, olive, lard and colza oils are all solid long before linseed oil, and the latter "thaws" much sooner than any of the others. The solidification point is more useful in identifying individual oils than in detecting sophistication. However, it may be laid down as a general rule that the freezing-point of drying oils is much lower than non-drying oils, and although fish oils in many of their properties resemble drying oils yet in this respect they show a marked difference, the freezing-points of most fish oils being about 0° C., whereas linseed oil is – 27° C.

SOLIDIFICATION POINT OF OILS.

Oil.	Massie.	Braconnot.	Château.	Fr. Chatin.	Various.
	Degrees C.	Degrees C.	Degrees C.	Degrees C.	Degrees C.
Sperm	—	+ 8	—	—	—
Olive	+ 2·5	+ 7	– 2·5	+ 2·75	+ 2 + 6
Whale	—	—	—	—	0 + 1 + 2
Cod-liver	0	—	—	—	1·0
Neatsfoot	0	—	—	—	1·5
Trotter	0	—	—	—	1·5
Lard	0	—	—	—	—
Arachis	+ 2	—	—	– 3	– 7
Cotton-seed	—	– 2	—	—	– 3
Rape	– 3·75	– 5	– 3·8	– 3·75	– 3·25 – 4
Sesame	– 5	—	—	—	—
Oleic Acid	– 6 to 7	—	—	—	—
Colza	– 6	– 6	– 6·3	– 6·25	– 6·75
Hazel-nut	– 10	– 10	– 18·5	—	– 20
Sunflower	– 16	—	—	—	—
Grape-seed	—	—	—	—	– 16
Black Mustard	– 1 to 2	—	– 17·5	—	0
Beech-nut	– 17	– 17	– 17·5	– 17·5	—
Castor	– 18	—	– 17·5	– 18	—
Poppy-seed	– 18	– 18	– 18·5	– 18	—
Camelina	– 18	– 18	– 18·8	– 18	—
Apricot	– 20	—	—	—	– 21 – 22
Almond	– 25	– 2·5 ?	– 2·3 ?	—	– 10 – 12
Hemp-seed	– 15	– 15	—	—	– 26·7
Linseed	– 15 to 20	—	– 27·5	– 27·6	—
Walnut	– 27	– 27·5	– 27·5	– 28	—
Pine	—	—	—	—	– 30
White Mustard	—	—	—	—	– 17·5

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SOLIDIFICATION POINT OF LINSEED OIL AND ITS FATTY ACIDS.

Linseed Oil.		Fatty Acids.			
		Solidification Point.		Melting Point.	
Degrees C.	Observer.	Degrees C.	Observer.	Degrees C.	Observer.
−16 after standing several days	Gusserow.	13·3	Von Huble.	17	Von Huble.
		17·5	Allen.	24	Allen.
		16 to 17	De Negri & Fabris.	Below 13	Dieterich.
−27	Château.			20 to 21	De Negri & Fabris.
Melts at −16 to 20	Glaesner.	19 to 20·6	Holde.		
		19 to 19·4			
		20·2 to 20·6	Lewkowitsch		

6. Colour Tests for Linseed Oil.

A. *The Sulphuric Acid Spot Test for Linseed Oil.*—This is undoubtedly the most valuable qualitative test for the purity of linseed and other oils. Yet it is conspicuous by its absence from most technical works on oils or is disposed of by a few words in passing. The apparatus required is simple: (1) Three or four glass rods rounded or dubbed; (2) a stoppered bottle containing concentrated sulphuric acid; (3) a white porcelain slab with several circular hollow depressions as shown in the illustration, or a plain porcelain slab will do, or even a plate of glass with a sheet of white paper underneath, but for the sake of cleanliness and accuracy the circular depression plate is much to be preferred as the oils have a tendency to run into one another and it is difficult to avoid getting into a nasty mess when testing a series of oils. The circular depression for each oil avoids all this unpleasantness. Rubber bulb tubes are also used for spotting acid.

Process.—Put a number on each sample bottle of oil to correspond with the number of the depression on the plate. Then spot ten drops of each oil to be tested from its bottle on to its corresponding depression on the plate. Then dip one of the glass rods, which must be scrupulously clean, into the sulphuric acid bottle and carefully carry away on it as much as will spot one drop of acid, being careful not to drop the superfluous acid about the table. Two drops are generally necessary, one above the other. Care has to be taken to place the rod in a beaker when done with. It must not be allowed to touch the oil. If it be desired to stir the oil another rod must be used. The two drops may often be spotted at once from the acid secured on the rod by one dip, but it is best to be content with one drop and to make a second dip into the bottle for the second drop. The rod must be allowed to drain, but not too much. If a Mohr's

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burette with a glass stop-cock is available, the sulphuric acid can be spotted from it with greater ease and cleanliness.

Pure linseed oil under the sulphuric acid spot test behaves as follows :—

The spot quickly assumes a yellowish tint, passing quickly to orange, then to bright red, darkening gradually to brownish black.

The oils which may be detected in linseed oil by the sulphuric acid spot test are :—

(1). *Rosin Oil.*—Should the linseed oil contain as little as 5 per cent. only of rosin oil the behaviour of the sample will show a marked deviation from the pure sample. It becomes brownish red instantly and passes much more quickly to black than the pure sample, and the black coloration is always deeper than that of the pure oil.

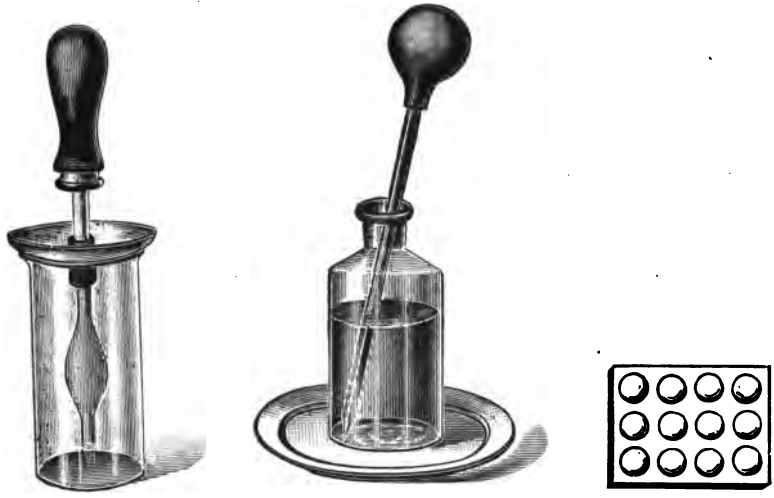

FIG. 25.—Apparatus for Detecting Rosin Oil in Linseed (Sulphuric Acid Spot Test).

Even smaller quantities than 5 per cent. of rosin oil can be detected by this means. If the smell, the taste, the gravity and the sulphuric acid spot test all point to the presence of rosin oil, it should now be determined by Allen's saponification and ether extraction method (see p. 112), and very possibly it will be the only adulterant present.

(2). *Whale Oil.*—As soon as the centre of a spot of pure whale oil is touched by the sulphuric acid a rapid motion takes place from the centre to the circumference, chasing, so to speak, a grey coloration in front of it. The central spot, at first red, passes to brown red, and, in a quarter of an hour's time, the whole of the spot becomes a brownish black. In testing linseed oil for whale or other oils in this way it is better to simultaneously test a standard sample of genuine linseed oil, a standard sample of genuine whale or other oil along with the oil to be tested, examining and comparing the effects.

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(3). *Cod Oil.*—The moment the drop of acid touches the oil red-violet striæ appear in the centre, which afterwards turn to dirty red and finally to black. The centre of the spot is dark yellow, passing to bright brown, the contour of the spot of oil is hardly altered. Another description says the effect of the acid is to give an orange spot with a dark nucleus giving birth to a purplish floating crust which quickly browns. Stirring gives a bluish purple, turning to red and finally to brown.

(4). *Skate-Liver Oil* (Density 0·928 at 15° C.).—Sulphuric acid spotted in the centre causes beautiful violet striæ to swim about, which suddenly pass to bright brown. The centre of the spot is red, covered with brown striæ. A dirty grey aureole surrounds the circumference of the spot. Gaseous chlorine does not darken the colour of this oil even after half an hour's contact.

(5). *Rape Oil.*—Before stirring gives a greenish-blue aureole. After stirring a bluish-green.

(6). *Hemp-seed Oil.*—The reagent gives a yellow spot where the acid has been dropped, the remainder of the oil turns greenish blue, then a beautiful emerald green, with small brown striæ on the edges.

(7). *Ravison Oil.*—This oil is known by its sharp pepper-like taste. The reagent gives a bluish-green aureole, which soon passes to dirty grey, leaving on the edges dark brown spots. The centre instantly becomes reddish yellow, turning to bright brown.

(8). *Sesame.*—Is the only oil which gives a characteristic bright red.

(9). *Poppy.*—Gives a yellow immobile spot with orange rings. Turns buff on stirring, and finally brown red with disengagement of gas.

(10). *Walnut Oil.*—Yellow spot, which moves from one side to the other, and gives birth to quite an orange pellicle on the surface of the oil; stirring produces dark streaks and liberates bubbles of gas.

(11). *Mustard Oil.*—According to Lefevre, gives a dark yellow spot with fibrous streaks, inclining to orange, and a nucleus which rapidly darkens. On stirring it gives a red-brown colour. On the other hand, according to Heydenreich, black mustard oil behaves somewhat like rape, giving a greenish blue both before and after stirring.

B. *Tetrabromide of Tin Test for Rosin Oil.*—Tetrabromide of tin, $SnBr_4$ (made by dropping dry bromine through a separating funnel into a well-cooled glass flask containing granulated tin), gives a characteristic violet-red coloration in presence of rosin oil. It is, perhaps, equally delicate and characteristic a test for rosin oil in linseed oil as the sulphuric acid spot test. This test is due to Allen, and is a modification of Renard's stannic chloride $SnCl_4$ test.

C. *The Liebermann-Storch Acetic Anhydride and Sulphuric Acid Coloration Test for Rosin Oil.*—Storch has adapted Liebermann's colour test for rosin acids for the detection of rosin oil in linseed and other oils. From 1 to 2 c.c. of the oil to be tested are agitated with

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1 c.c. of acetic anhydride with the aid of a gentle heat. After settling and cooling the acetic anhydride is drawn off by a pipette and one drop of concentrated sulphuric acid added. A fine but fugitive violet coloration is imparted to the acetic anhydride if rosin oil be present in the oil tested. This test is especially applicable for testing mixtures of rosin oil and mineral oil. Morawski obtained the undernoted colorations with the following oils :—

Oil.	Coloration.	Oil.	Coloration.
Olive - -	Light Green.	Palm - -	Brownish Yellow.
Sesame -	Greenish Blue Gradual.	Bone Fat Acids -	Brownish Yellow.
Hemp - -	Green.	Whale Stearine -	Brownish Yellow.
Linseed -	Green.	Olein - -	Brownish Yellow.
Cotton -	Green.	Crude Olive Acids -	Light Brown to Dark Green.
Arachis -	Red Brown.	Herring -	Cherry Red to Brownish Black.
Rape - -	Greenish Yellow.	Sunflower -	Blue Violet to Blue.
Castor -	Yellowish.		
Cocoa-nut -	Yellowish.		
Palm-nut -	Yellowish.		
Beef Tallow	Yellowish.		

These colorations do not as a rule interfere with the detection of rosin oil.

7. *Flash Point of Linseed Oil, Gray's Apparatus (Closed Test).*

Description.—This apparatus (Fig. 26) consists of a brass oil-cup, *a*, of the same dimensions as the oil container used in Government Standard Petroleum Testers (Abel's). On this cup, which is set on a suitable stand, is placed a tight-fitting cap or lid. Through the lid a steel shaft passes, carrying on the top a small bevelled wheel, *h*, the bevel of which is milled, and on the lower part two sets of stirrers, one of which is below the surface of the oil, and the other in the vapour space above. A horizontal shaft carried on two standards terminates at one end in a bevelled wheel, *g*, which gears with the wheel *h*, and at the other end in a disc, *c*, the outer rim of which is made of bone or other suitable non-conductor of heat, and on this disc is fixed a handle, *b*, for rotating the shaft. This horizontal shaft carries a collar, *e*, from which there projects two small pins at diametrically opposite points. By sliding the shaft slightly to the right the bevelled wheels are drawn out of gear, and the pins projecting from the collar put in position for actuating the testing arrangement. On the lid proper there are three orifices, one immediately in front of the test-light, *d*, and the other two at either side of it. Above the lid is the sliding cover, *s*, in which there are two orifices corresponding to those on the fixed lid. When the bevelled wheels are in gear the sliding cover

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is held in its normal position by the spring at *p*, and the orifices in the fixed lid are covered. When the sliding cover is moved by bringing the pin *e* in contact with the pin *p* and turning the disc, *c*, about a quarter of a turn round, the orifices in it coincide with those on the fixed lid, and at the same time the test-light is depressed and brought into position for producing the flash when the proper temperature has been reached.

In places where gas is not available, a good substitute to use for the test-flame may be obtained by passing a current of air or hydrogen through cotton-wool or other convenient medium saturated with gasoline or other light hydrocarbon spirit.

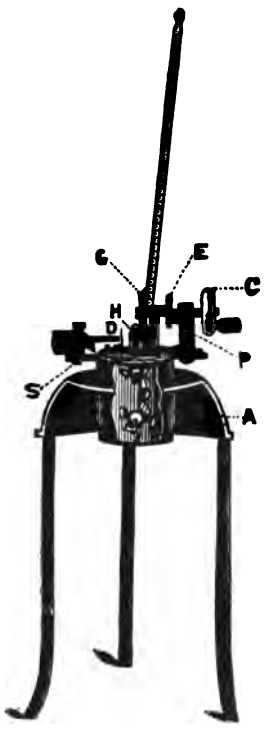

FIG. 26.

FIG. 27.

Fig. 26.—Gray's Flash Point Apparatus for Linseed, etc., Oils (Closed Test).
Fig. 27.—Apparatus for Determining Flash Point of Linseed Oil (Open Test).

Process.—Fill the oil-cup, *a*, up to the mark inside with the oil to be tested, replace the cap, and insert the thermometer in the tube provided for it. Apply a light to the heating lamp, the heat from which may be applied either direct or through the medium of a convenient sand bath. Light the test-jet also, and adjust flame to about $\frac{1}{8}$ of an inch

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in length. During the heating of the oil the stirrers should be moved at short intervals by turning the handle, *b*, several times. The more rapidly the oil is heated the oftener the stirrers should be rotated. When nearing the point at which the oil is expected to flash, it is advisable to reduce the rate of heating in order that the observations may be more readily and accurately made. If necessary, a rough test may be performed first, to give some indication of the temperature at which the oil is likely to flash. Observations may be taken every degree or half degree, a few turns of the stirrer being given between each test. The rate of heating is immaterial provided it is not too rapid to allow of readings being taken. A convenient time is ten to twenty minutes.

8. Index of Refraction.

The index of refraction has not been much used as a means of identification or for detection of sophistication in oils. The following results are given for the purpose of comparing that of linseed oil with other oils :—

Index of Refraction of Linseed Oil :—

At 15° C. 1·4835 to 1·4855 (Strohme).
At 60° C. 1·4660 (Thorner).

Index of Refraction of Fatty Acids of Linseed Oil :—

At 60° C. 1·4546 (Thorner).

Index of Refraction of Certain Oils.

Index determined at 15°, correction 0·00037 at 15° C.

Earth-nut, refined	1·47325	Trotter	1·47045
,, ,, crude	1·47315	Horse-foot	1·47095
Olive, Tunis	1·47215	Castor	1·47990
,, Kabylie	1·47015	Almond, sweet	1·47410
,, edible	1·47130	Cotton	1·47440
,, fine	1·47070	Black Mustard	1·47490
Walnut	1·47160	Lard (oil)	1 47195
Poppy	1·47730	Oleic acid (sap.)	1·46245
Sesame, crude	1·47490	Whale (St. Vincent)	1·47505
,, refined	1·47400	,,	1·47925
Linseed	1·48140	Cod-liver, Hogg	1·48600
Neatsfoot	1·47550	,, blonde	1·48335

The following are indices of refraction at 15° C. of linseed oil, and of substances which may be present in it as adulterants or otherwise :—

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Oil.	Refractive Index.	Oil.	Refractive Index.
Linseed	1·484-1·488	Turpentine	1·464-1·474
Cotton-seed	1·475	Rosin (Colophony)	1·548
Rosin oil	1·535-1·549	Maize	1·4765
Mineral	1·438-1·507	,,	1·478 (at 20° C.)
Fish	1·480		

REFRACTION OF PURE AND IMPURE DRYING, ETC., OILS WITH JEAN'S REFRACTOMETER.

Raw linseed oil—Laboratory standard sample	+ 53°
Refined raw linseed	+ 49°
Raw linseed + 20 per cent. rosin oil	+ 67°
,, + 20 ,, hemp-seed oil	+ 47°
,, + 20 ,, mineral oil	+ 17°
Poppy (*œillette*) oil	+ 29°
,, ,, old	+ 35°
Poppy (*pavot*) Calcutta oil	+ 27·5°
,, ,, ,, old	+ 33°
Hemp-seed oil	+ 30° to 32°
Walnut ,,	+ 36°
Ravison ,,	+ 25°
Colza ,,	+ 17° to 18°
Rosin [1] ,,	+ 78°

REFRACTION OF PURE AND IMPURE LINSEED OIL WITH HEFELMANN'S REFRACTOMETER.

	Refractometer Number at 200° C.
Pure Linseed Oil (raw)	80·5 to 82·2
,, (boiled)	80·5 to 84·2
Linseed Oil + 20 per cent. rosin	above 100·0
,, + 10 ,, ,,	94·5
,, + 5 ,, ,,	88·4
,, + 20 ,, rosin oil	above 100·0
,, boiled + 20 per cent. rosin oil	above 100·0
,, ,, + 10 ,, ,,	92·7
,, ,, + 5 ,, ,,	88·2
,, ,, + 15 ,, cotton-seed oil	78·7
,, ,, + 15 ,, rape oil	79·1
,, + 10 per cent. mineral oil	89·6
,, boiled + about 30 per cent. of manganese rosinate	84·9

[1] With some samples the whole field of the refractometer is black.

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PURE AND IMPURE BOILED LINSEED OIL—CHEMICAL AND PHYSICAL DATA OF.

	Refraction at 25° C.	Polarisation.	Iodine number.	Saponification number.	Saponification (turbidity) test.
Litharge boiled oil 1	84·1	feebly −	163·0	188·9	clear
,, ,, 2	84·2	,,	167·1	—	,,
,, ,, 3	84·1	,,	165·0	—	,,
,, ,, 4	82·8	,,	167·5	188·4	,,
,, ,, 5	82·9	,,	162·3	188·7	,,
Manganese boiled oil 1	80·6	,,	172·1	188·9	,,
,, ,, 2	84·0	,,	170·4	186·1	,,
,, ,, 3	82·9	,,	167·7	190·2	,,
,, ,, with 3 p.c. rosin oil 4	84·9	± 0	164·8	185·4	slightly turbid
Pure linseed oil	80·6-84·2	feebly −	160-180	180-190	clear
Litharge boiled oil 6	95·8	strongly +	143·6	160·8	quite turbid
,, ,, 7	96·3	,,	158·3	169·2	,,
,, ,, 8	89·6	,,	154·7	169·3	,,
,, ,, 9	91·9	,,	153 4	167·5	,,
Manganese boiled oil 5	90·0	,,	161·1	169·3	,,
,, ,, 6	over 100·0	,,	129·8	169·6	,,
Boiled oil + 20 p.c. rosin	,, 100·0	slightly +	acid number 45·6	197·6	slightly turbid

Therefore, if a sample of raw or boiled linseed oil, examined at 25° C., is found to give a refractometer number greater than 84·5, it is open to the suspicion of containing an admixture of rosin, rosin oil, metallic rosinates, or mineral oil. On the other hand, if the refraction falls below 80, then additions of slightly or non-drying fatty oils must be looked for. Very small percentages are not detected by the refractometer, but such are not likely to occur in practice.

The above table shows in how far the refractometer test coincides with the results of chemical analysis. All oils exhibiting normal re-

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fractions were found pure, and those refracting abnormally adulterated. The foregoing were purchased as pure boiled oil, with the exception of the last, containing 20 per cent. of rosin, which was prepared in the laboratory. Out of fifteen commercial samples, nine were pure and six contained rosin oil, the only adulterant detected.

A slight turbidity on the addition of water to the saponified oil (p. 112) indicates the presence of small admixtures of rosin or metallic rosinates. (The production of a dark, flocculent, heavy precipitate in the case of manganese boiled oil should be disregarded as common to all boiled oil of this class. It cannot, therefore, be considered as indicative of the presence of rosin oil or mineral oil.) The polarisation test gives either neutral or slightly dextro results, and both saponification and iodine numbers fall within the ordinary limits, so that no differentiation between slight quantities of added rosin or metallic rosinates is possible. The saponification test is reliable in presence of rosin oil or compounds of rosin acid with metallic oxides and mineral oils, but isuncertain when rosin is present.

9. Polarimetric Tests.

The investigations of Bishop and Peters on the opticity of a number of oils show that, with the exception of castor oil, croton oil and rosin oil, the only dextro rotations are produced by sesame (high) and olive oil (feeble), all the others, including linseed oil, being either optically inactive or having a slight levo rotatory power.

TABLE SHOWING THE ACTION OF OILS ON POLARISED LIGHT.

Oil.	Saccharimetrical Degrees (Peters).	Saccharimetrical Degrees (Bishop).
Almond (sweet)	−0° 2″	−0° 7″
Beech-nut	Inactive.	—
Castor	+40° 7″	—
Colza (European)	−1° 2″	−2° 1″
„ (Japanese)	—	−1° 6″
Cotton-seed	−0° 7″	—
Croton	+42° 5″	—
Earth-nut	+0° 5″	−0° 4″
Hazel-nut	−0° 2″	—
Hemp-seed	−0° 5″	—
Linseed	−0° 3″	−0° 3″
Olive	+0° 75″	+0° 6″
„	+1° 3″	—
Poppy-seed	+0° 75″	—
Sesame	+5° 0″	+3° 1″
„ (warm)	—	+7° 2″
„	—	+4° 7″
„	—	+3° 9″
„	—	+9° 0″
„ (Indian)	—	+7° 9″
Walnut	—	−0° 3″

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(1). *The Polarimetric Examination of Linseed Oil Sophisticated with Refined Rosin Oil (R.R.O.).*—According to Aignan such a mixture rotates the plane of polarisation to the right by an angle perceptibly proportional to the quantity of rosin oil which it contains. If the rotation observed with a 200 millimetre tube be represented by [a]D and the weight of the rosin oil in 100 parts by weight of the mixture by h, we get in the case of a mixture of linseed oil with—

1.	Refined rosin oil	$[a]D = + \frac{14}{15} h.$
2.	Choice white rosin oil	$[a]D = + \frac{17}{15} h.$
3.	Rectified rosin oil	$[a]D = + \frac{21}{15} h.$

The first mixture is the most common. In actual practice, therefore, all that has to be done is to measure [a]D by the polarimeter, and to estimate h as refined rosin oil, according to the formula $h = [a]D \frac{15}{14}$. The oils in question being dark in colour, it is better to work in a 100 millimetre tube and to calculate

$$h = [a]D = \frac{15}{7}.$$

(2). *Estimation of Rosin Oil in Paint by the Polarimeter.*—A. A certain amount of the paint is frequently stirred and shaken up with ether and allowed to settle. The ether containing the oil in solution floats to the surface and the polarisation tube is filled with the ethereal solution. If no optical deviation be produced, there is no rosin oil in the paint tested. On the other hand, if [a]D be the rotation towards the right with a 200 millimetre tube, according to Aignan's researches on the rotatory power of an ethereal solution of linseed oil containing rosin oil, the proportion of rosin oil may be calculated by the formula—

$$h = \frac{a[D]}{43^2}$$

B. A known weight p^1 of the ethereal solution is run into a flask and heated on the water bath at 100° C. (212° F.) so as to drive off the ether; the oil which boils only at 300° C. (572° F.) is left in the flask. Let its weight be represented by p^2. The proportion $\frac{p^1}{p^2} 100 = h^1$ per cent. of oil (linseed oil and rosin oil) contained in the ethereal solution examined by the polarimeter. If $h^1 = h$, it may be taken for granted that the paint contained linseed oil free from rosin oil. Generally, h^1 is greater than h, then $\frac{h}{h^1} 100$ will give the percentage of rosin oil contained in the linseed oil which was used to make the paint.

10. *The Percentage of Spirits of Turpentine and other Volatile Oils, Benzene and other Solvents, etc.*—Spirits of turpentine, coal tar, naphtha (benzol and its homologues), shale naphtha and petroleum naphtha (shale spirit deodorised [*sic*], petroleum spirit, etc.) are

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readily detected in linseed oil, whether raw or boiled, and whether they be present in comparatively small or relatively large proportions, by their characteristic smells, which are accentuated by placing the oil in a test tube or other vessel so as to half fill it, and then dipping the tube into boiling water after having corked it up. On releasing and removing the cork the characteristic odour of the individual volatile solvent present will make itself felt.

Moreover, the greatly diminished flash point will also show substances of this nature, and also the gravity if present in any quantity. To estimate the volatile oil quantitatively about 300 grammes are heated by a bath of molten paraffin wax or by an air bath to about 130° C. in a flask capable of being connected with a vessel in which steam is generated. It therefore has a cork fitted with an inlet tube reaching nearly to the bottom of the vessel, a thermometer inserted into the oil, and a tube for leading the steam and the vapour of spirits of turpentine, etc., which it carries in its train to a Liebig's condenser. The distillate will consist of two layers, an upper one of spirits of turpentine, etc., floating on a layer of water. The former may be separated from the latter by a separating funnel, and weighed or measured. The layer of condensed water retains traces of the volatile oil, but so small as may very well be neglected. McIlheny found it to be about $\frac{1}{3}$ of 1 per cent.

Petroleum naphtha may be separated from spirits of turpentine by treating the mixture with fuming nitric acid. Petroleum naphtha remains unattacked, but spirits of turpentine is converted into substances which can be dissolved in water, and thus eliminated from the mixture, leaving a residue of more or less pure petroleum naphtha. A measured amount of the distillate is run into 300 c.c. of fuming nitric acid (very slowly drop by drop) contained in a 750 c.c. flask attached to a reflux condenser. As each drop of the oil falls on the fuming acid a very violent reaction ensues, and the flask should be immersed in cold water to keep it cool, and it should be agitated from time to time. When the whole of the volatile oil to be treated has been added the flask is allowed to stand until all action has ceased, when its contents are run into a separating funnel (Fig. 28) and repeatedly washed with hot water, which eliminates the products produced by the interaction of the spirits of turpentine and the fuming nitric acid, such products being soluble in water. The residual petroleum oil is now measured and brought to per cent. by volume of the distillate and then to per cent. of the original oil. However applicable this method may be to petroleum naphthas consisting of pure paraffinoid hydrocarbides, it is on the face of it wholly inapplicable to *inter alia* shale naphtha, largely consisting of members of the ethylenic series of hydrocarbides, the so-called "olefins". This is a point which seems to have been unaccountably overlooked by those writers who recognise and quote the above method as an efficient one for separating mineral naphtha from spirits of turpentine.

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11. *Determination of Rosin Oil and Mineral Oil in Linseed Oil by Saponification and their Extraction from the Solution of the Resultant Soap by Ether.*—Five grammes of the sample to be tested are saponified with 25 c.c. of alcoholic potash, 80 grammes of caustic potash to the litre, in a capacious porcelain basin, care being taken to apply heat very cautiously at first, as the mixture froths much and is apt to take fire. When frothing has ceased a greater heat may be applied, but in bringing the soap to dryness at the end care must be taken to avoid charring or overheating which might distil off some of the rosin, etc., oil. The operation may be done very well on the sand bath by the exercise of care. It is advisable to have a clean iron or copper plate to place on the top of the porcelain basin to extinguish the flame should its contents catch fire. The resultant soap is dissolved in the basin in 50 c.c. of boiling water and transferred to a separating funnel of about 200 c.c. capacity, using about 20 to 30 c.c. of water for rinsing out the basin. (See p. 109.) After cooling 50 c.c. of ether are added and the solution and ether thoroughly shaken in the funnel. The funnel and its contents are allowed to stand, when the ether separates out as an upper layer containing the greater portion of the rosin oil. A few drops of alcohol hasten the separation. The soap solution is then run off through the stop-cock whilst the ethereal solution is run into a flask. The soap solution is then returned to the funnel and again agitated with ether and the process repeated until the soap solution cedes no more rosin, etc., oil to the ether. The ethereal solutions are united and washed with a small amount of water to eliminate any dissolved soap. The ether is then distilled off on the water bath and the residue dried and weighed. In a works laboratory the greater part of the ether may be driven off by using several beakers of boiling water brought from the waste steam pipes, and if the laboratory lights are all out there is no danger of explosion. According to two French authors $\frac{9}{10}$ of the laboratory explosions are ether explosions. Ether vapour will explode or burn 12 feet away from its source, and the flame or explosion strike back to it. Glacial acetic dissolves mineral but not rosin oil.

FIG. 28.—Separating Funnel showing Separation of the Ethereal Solution of Rosin Oil from the Soap made from Linseed Oil containing Rosin Oil.

12. *Estimation of the Free Acid in Linseed Oil.*—Pure samples of

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linseed oil, unless oxidised or thickened or very old, contain but small amounts of free fatty acid, whilst if sophisticated with rosin oil, which abounds in free rosin acids, the percentage acidity may be much increased. In the distillation of rosin there is a good deal of vesicular carrying over of unchanged rosin in the train of the vapour of rosin oil, hence the marked acidity of the latter. But the natural acidity of linseed oil increases greatly with age. So that although a high acidity might indicate the presence of rosin acids, and hence of rosin oil or free rosin, yet it would not be safe to come to a decided conclusion without confirmatory evidence. A portion of the acidity may even be due to free sulphuric acid used in refining the oil, but any appreciable proportion would point to unmitigated carelessness on the part of the refiner in washing, seeing he hardly ever uses more than about 1 per cent. of acid. The actual amount of acidity due to sulphuric acid is estimated by repeatedly boiling a known quantity of the oil with water until free from acidity, collecting the wash waters, concentrating, and, after cooling, adding neutral potassium iodide and iodate and titrating the liberated iodine, if any, with sodium hyposulphite. The free iodine found is calculated to caustic potash, KHO, and deducted from the potash required to neutralise both the sulphuric acid and the organic acids present in the sample. The remainder gives the free organic acids, which consist of the fatty acids from linseed oil alone if the sample be pure, but if adulterated it may represent in addition the acidity due to the presence of rosin added either as such or in the form of rosin oil. The total free acid is determined as follows: From 5 to 10 grammes of the oil are weighed into a flask and 50 c.c. of alcohol added; if methylated spirit be used it must be previously neutralised, as it is always acid. Heat to boiling on the water bath, shake and titrate with semi-normal alkali, and the result is calculated to milligrammes of caustic potash, KHO, per gramme of oil.

The acidity of linseed oil expressed as oleic acid, according to Nordlinger, varies from 0·41 to 4·19. McIlheny gives an acid value of 3·0 as the usual figure, which equals oleic acid 1·51. A very old sample gave 7·1. Mills allows a maximum acid value of 10. It will be readily understood that boiled oil gives higher acid figures than raw oil. Weger allows as high an acidity as 12, and even in very much thickened oils he allows 30. Here, however, it may be noted that free rosin may be introduced by the addition of fused metallic rosinates, the so-called "resinates," with the accent on the *e*. The acid figure of boiled oil, says McIlheny, is usually below 5, but it is more uncertain than the raw oil. A figure above 10 is in his opinion undoubtedly due to the presence of rosin, which seems rather a dogmatic dictum. The acid value of rosin varies from 145 to 180, and its detection is facilitated by its giving both a high bromine substitution figure as well as an addition figure. But the safest method is to estimate it quantitatively by one or other of the methods adopted in soap analysis, such as Gladding's or Twitchell's. But the process

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often recommended to exhaust the oil with *pure alcohol* and then to add an alcoholic solution of basic acetate of lead is not reliable, as linoleate of lead formed from the free linoleic acid does not dissolve in alcohol to any very appreciable extent more than the rosinate of lead. The operator is thus thrown back upon the character of the precipitate—always a very unreliable indication.

It is quite unnecessary to give a table of acid values of linseed oil as they are of no use without the history of the samples to which they refer.

13. *Saponification Value of Linseed Oil.*—This is defined as the number of milligrammes of caustic potash, KHO, required to completely saponify 1 gramme (*i.e.*, 1,000 milligrammes) of linseed oil. Or, to use British units, it is the number of grains of caustic potash required to saponify 1,000 grains; or, for that matter, the number of tons of caustic potash required to saponify 1,000 tons of oil. Divided by 10, the saponification value again gives the number of pounds of caustic potash required to saponify 100 lb. of oil. It is really a very simple matter, but the use of foreign, if scientific, units (grammes per litre when pounds per 100 gallons has the same meaning and effect) tends to render it unintelligible to the non-scientific layman.

Reagents.—(1) Standardised hydrochloric acid, the strength of which is expressed in terms of caustic potash, KHO. The semi-normal acid is the most suitable; such an acid contains in every litre, that is, in every 1,000 c.c., 18·25 grammes of anhydrous hydrochloric acid, HCl. The pure solution of hydrochloric acid is diluted by referring to the tables of density with distilled water until this strength is reached; its exact strength is determined by estimating its chlorine as chloride of silver, AgCl. This solution may also be standardised by titration with a known weight of pure, dry, freshly ignited sodium carbonate, Na_2CO_3, every 106 grammes of which are equal to 112·2 grammes of caustic potash, KHO. The hydrochloric acid, to be perfectly semi-normal, should be so diluted that 0·265 grammes of pure anhydrous sodium carbonate require 10 c.c. of the diluted acid for neutralisation. The exact strength of the standard hydrochloric must always be known in terms of caustic potash, which, when exactly semi-normal, is 1 c.c. = 0·02805 KHO.

Alcoholic Potash Solution.—This need not be of a very exact predetermined strength, but should approach, as far as practicable, the semi-normal, so that 1 c.c. of the alcoholic potash solution neutralises 1 c.c. of the semi-normal acid. To 1 litre of alcohol 30 to 40 grammes of caustic potash are added, or rather 75 c.c. of an aqueous solution of caustic potash of 45° Baume or 90 c.c. of 36° Baume are run into a litre of alcohol, and the whole allowed to stand and filtered through a ribbed filter, or decanted after clarifying into a bottle with a straight neck and corked with an indiarubber stopper, through a hole in which a 25 c.c. pipette passes, the end of which is closed by a piece of indiarubber tubing and a small clip. It is not advisable to prepare too much of the solution as it is not stable. The alcohol should be the

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purest obtainable, but if methylated spirit be alone available it should be purified by redistilling it over soap. The alcohol should not become brown, the colour of rum or cod oil in the process. A yellowish tint does not interfere with the titration.

Process.—From 1·5 to 2 grammes of the filtered oil are weighed exactly into a 150 to 200 c.c. flask, then 25 c.c. of the alcoholic potash solution measured out by the pipette in the stopper of the bottle are added. It is not necessary to rigidly measure out 25 c.c. exactly, but precisely the same quantity must be taken for each experiment. After the contents of the pipette have drained it is better to grasp tightly the bulb of the pipette in the palm of the hand and "squeeze," as it were, the remainder of the liquid out of the pipette; the heat of the hand expands the air inside the pipette, and the top aperture being closed by the finger, the expansion of the air causes all the liquid, except the merest traces, to be forced out of the mouth of the pipette. A similar flask with 25 c.c. of alcoholic potash is used as a blank experiment, and both are heated on the water bath simultaneously for half an hour, care being taken to place a small watch glass or funnel on the mouth of each of the flasks. Two to three drops of phenol-phthalein are then added to each flask, and the contents titrated with the semi-normal hydrochloric acid. The difference between the blank flask and that containing the oil is calculated into potash; the volumes of alkaline solution used being equal, the difference is the potash used up by the oil. 1 c.c. of acid = 0·02805 grammes of KHO, and this factor multiplied by the number of c.c., gives a number which, when reduced to 1 gramme of the oil, gives the number of milligrammes of caustic potash consumed by 1 gramme of oil, and this figure is generally called the Koettstorfer figure or saponification value.

EQUIVALENT WEIGHTS OF CAUSTIC AND CARBONATED ALKALI.

Potash Alkali.	Formula.	Molecular Weight.	Equivalent Weight.	Soda Alkali.	Formula.	Molecular Weight.	Equivalent Weight.
Caustic Potash -	KHO	56·1	112·2	Caustic Soda -	NaHO	40	80
Anhydrous Potash	K_2O	94·2	94·2	Anhydrous Soda -	Na_2O	62	62
Potassium Carbonate - -	K_2CO_3	138·2	138·2	Sodium Carbonate	Na_2CO_3	106	106
				Soda Crystals -	$Na_2CO + 10aq$	286	286

The strength of commercial alkali is always expressed in per cent. of anhydrous alkali, as in the second horizontal line; if the caustic potash be pure it contains 94·2 per cent., and knowing the saponification value of the oil and the percentage of anhydrous potash in the alkali being used the calculation of the amount required to saponify any given oil or rosin whose saponification value is known is a simple matter.

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Analytical Examples.—(1) Where the acid and alkali are both semi-normal, and (2) where neither are semi-normal.

Example 1.—Linseed oil taken - - - 1·75 grammes.
½ Normal alcoholic potash taken - 25 c.c.
½ Normal acid used in back titration 13 c.c.
12 c.c.

Each c.c. normal acid = 0·02805 grammes KHO.
12 ,, ,, = 0·33660 ,, ,,
0·3366 grammes KHO = 336·6 milligrammes KHO.

$$\frac{336{\cdot}6}{1{\cdot}75} = 192{\cdot}4 \text{ milligrammes KHO.}$$

It therefore took 192·4 milligrammes of caustic potash to completely saponify 1 gramme of the linseed oil in question.

Example 2.—Benedikt and Ulzer give the following example: 2·012 of oil were saponified with 25 c.c. of alcoholic potash and 9·65 c.c. of standardised hydrochloric acid were used in back titration. 25 c.c. of the alcoholic potash = 22·5 c.c. of the standard hydrochloric acid. Again 1 c.c. of the standard hydrochloric acid = 0·0301 gramme caustic potash, KHO. There was used in saponification of the oil, therefore, an amount of caustic potash equal to 22·5 − 9·65 = 12·85 c.c. of test acid, hence 12·85 × 0·0301 grammes = 386·8 milligrammes, for 2·012 grammes = 192·24 milligrammes for 1 gramme of oil. The oil tested therefore had the saponification value of 192·24.

The saponification value of linseed oil is higher than that of most oils, but it can only be regarded as affording an indication of purity. Even a comparatively high saponification value is no guarantee that the oil is not adulterated with non-drying oils, which approach it very closely in this respect; neither is it a guarantee that unsaponifiable substances, such as hydrocarbide oils, are absent. The only way to determine the absence or presence of these is to actually separate them out (and weigh them) by the saponification process first introduced by Thomson, and afterwards elaborated by Allen, which consists in first saponifying the oil and then extracting the hydrocarbide oils from the aqueous solution of the resultant soap by ether. (See Fig. 28, p. 112, and context.)

SAPONIFICATION VALUE OF LINSEED OIL.

Saponification Value.	Observer.	Saponification Value.	Observer.	Saponification Value.	Observer.
189-195	Allen	190-195	Holde	187	McIlheny
195·2	Moore	190-192	Thorner	187·6	Filsinger
187·6	Dietrich	190-192·7	De Negris and Fabris	192·4	—

Saponification Value of Fatty Acids from Linseed Oil—198·8 (Dietrich).

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When the saponification value is as low as from 180 to 185 then adulteration with rosin oil or mineral oil is to be strongly suspected, and when it falls lower than 175 their presence is certain (Weger). It may fairly be demanded of a raw oil that its figure shall not be lower than 187 and of a boiled oil not lower than 186 (McIlheny). The high temperature used in the old-fashioned process of oil-boiling reduced the saponification value much greater than the much lower temperature now used with modern processes. The amount of metal introduced into the oil in the form of a metallic rosinate or linoleate is so small as not to produce any very appreciable effect on the saponification value, and some of the boiled oils now in the market have almost as high a saponification value as that of the raw oil.

14. *Iodine and Bromine Value of Linseed Oil.*—Linseed oil consists largely of unsaturated glycerides, the fatty acids of which combine by *direct addition* with 2, 4 or 6 atoms of bromine or iodine. Rosin, rosin oil and mineral oil do so only to a slight extent, and, menhaden oil excepted, no other adulterant to as great an extent as linseed oil itself. *Direct halogen addition* is not the only action; there is another in which one-half of the halogen combines with the oil and the other half combines with hydrogen, which the first half of the halogen has displaced from the oil. The halogen has formed a *substitution* compound with the oil. If the hydrogen of glycerides can be but very sparingly replaced by bromine or iodine, it is not so with rosin, rosin oil and mineral oils. In fact though rosin and rosin oil absorb large amounts of bromine they do so by substitution and not by direct addition, and with American petroleum oils substitution compounds bulk largely in the absorption.

The halogen absorption of oils is generally determined by Hübl's method. It affords valuable data as to purity of any sample of linseed oil. But it does not differentiate between absorption by (1) addition and (2) substitution. It fails to discriminate between rosin and linseed oil, the absorption figures of both linseed oil and rosin being somewhat close.

Reagents—(1) *Iodine Solution.*—Dissolve 25 grammes of pure iodine in 500 c.c. of 95 per cent. alcohol. Dissolve 30 grammes of mercuric chloride in 500 c.c. of 95 per cent. alcohol. The last solution, if necessary, is filtered, and then the two solutions mixed. The mixed solution should be allowed to stand twelve hours before using. (2) *Decinormal Hyposulphite of Sodium Solution.*—Take 24·6 grammes of chemically pure hyposulphite of soda freshly pulverised as finely as possible and dried between filter or blotting paper. Make this up to 1,000 c.c. at the temperature at which the titrations are to be made. (3) *Starch Paste.*—1 gramme of starch boiled in 200 c.c. of distilled water for ten minutes and cooled to room temperature. (4) *Solution of Iodide of Potassium.*—150 grammes of iodide of potassium dissolved in water and made up to 1 litre. (5) *Solution of Bichromate of Potassium.*—Dissolve 3·874 grammes of chemically pure bichromate of potassium in distilled water, and make the volume up to

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1 litre at the temperature at which the titrations are to be made. (6) *Chloroform.*—This should be pure and should not affect the titration results of the iodine solution after three hours' standing.

Manipulation—Standardising the Hyposulphite of Sodium Solution.—Run 20 c.c. of the bichromate of potassium solution to which has been added 10 c.c. of the solution of iodide of potassium into a glass-stoppered flask. Add to this 5 c.c. of strong hydrochloric acid. Allow the solution of hyposulphite of sodium to flow slowly into the flask until the yellow colour of the liquid has almost disappeared. Add a few drops of the starch paste and with constant shaking continue to add the hyposulphite of sodium solution until the blue colour just disappears. The number of cubic centimetres of hyposulphite solution used multiplied by 5 is equivalent to 1 gramme of iodine. *Example.*—20 c.c. $K_2Cr_2O_7$ solution required 16·2 c.c. hyposulphite of soda; then 16·2 × 5 = 81 = number of cubic centimetres of hyposulphite solution equivalent to 1 gramme of iodine. Then 1 c.c. hyposulphite solution = 0·0124 gramme of iodine. Theory for decinormal solution of hyposulphite of sodium, 1 c.c. = 0·0127 gramme of iodine. *Weighing the Sample.*—About 0·15 to 0·18 gramme of oil is to be weighed in a glass-stoppered flask holding about 500 to 800 c.c. *Absorption of Iodine.*—The oil in the flask is dissolved in 10 c.c. of chloroform. After complete solution has taken place 30 c.c. of the iodine-mercuric chloride solution is added from a pipette. The chloroform and iodine should give a clear solution; if not, more of the former must be run in for that purpose, and 25 c.c. more iodine if the colour be discharged, as an excess is necessary. After two hours the deep brown colour must still be persistent. The flask is now placed in a dark place and allowed to stand, with occasional shaking, for another two hours. *Titration of the Unabsorbed Iodine.*—300 to 500 c.c. of distilled water is added to the contents of the flask, together with 20 c.c. of the iodide of potassium solution. Any iodine which may be noticed upon the stopper of the flask should be washed back into the flask with the iodide of potassium solution. Any red precipitate of mercury iodide would indicate an insufficiency of potassium iodide which has to be remedied by adding more. The excess of iodine is now taken up with the hyposulphite of sodium solution, which is run in gradually, with constant shaking, until the yellow colour of the solution has almost disappeared. A few drops of starch paste are then added and the titration continued until the blue colour has entirely disappeared. Toward the end of the reaction the flask should be stoppered and violently shaken, so that any iodine remaining in solution in the chloroform may be taken up by the iodide of potassium solution in the water. A sufficient quantity of hyposulphite of sodium solution should be added to prevent a reappearance of any blue colour in the flask for five minutes. *Control Experiments.*—At the time of adding the iodine solution to the oil two blank flasks of the same size and nature as those used for the determination should be employed for conducting the operation described above without the presence of any

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oil. The difference between the mean of the results obtained by the blank experiments and that on the oil corresponds to the iodine used, which is brought to per cent. of the oil. This figure gives the iodine value. The iodine value of boiled oil (see p. 108) often approaches that of the raw oil, especially those with a rosinate or linoleate drier.

IODINE VALUE OF RAW LINSEED OIL.

Iodine Value.	Observer.	Iodine Value.	Observer.
171-175	Lewkowitsch.	173·5-187·7	Thomson & Ballantyne.
170-181	Benedikt.	171-179	Shukoff.
173-195	Ulzer.	176·3	Wijs, by his Iodine Chloride Method.
171-190	Holde.	201·8	

IODINE VALUE OF FATTY ACIDS OF RAW LINSEED OIL.

Iodine Value.	Observer.	Iodine Value.	Observer.
178·5	Williams.	159-185	De Negri and Fabris.
179-182	Lewkowitsch.	179-192	Holde.

IODINE VALUE AND OTHER "CONSTANTS" OF DIFFERENT BRANDS OF RAW LINSEED OIL.—(LEWKOWITSCH.)

Linseed Oil from	Sp. gr. at 15·5° C. (Water at 15·5° C. = 1.)	Acid No.	Saponification Value.	Iodine No.	Unsaponifiable Matter, per cent.	Oxidised Acids, per cent.
Finest Calcutta Linseed, two months old	0·9316	1·3	193·2	170·46	0·6	0·65
Finest Calcutta Linseed, three years old, kept throughout that period in an air-tight vessel, shielded against light	0·9324	1·3	192·5	174·03	0·7	0·70
Finest St. Petersburg Linseed, three months old	0·9334	1·3	192·2	177·25	1·1	0·88
Finest St. Petersburg Linseed, seven months old	0·9345	1·3	193·1	176·23	0·98	0·56
Baltic Linseed, commercial; the seeds contained an admixture of ravison and camelina seeds	0·9343	1·3	194·3	170·05	1·1	0·73
Finest and purest Baltic Linseed; the sample was kept away from light for thirteen years	0·9410	7·2	195·2	175·84	1·1	1·95

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IODINE AND SAPONIFICATION VALUE OF PRINCIPAL OILS.

Oil.	Iodine Values.			Saponification Values.		
	Min.	Max.	Average.	Min.	Max.	Average.
Candle-nut	136·3	163·7	150	184	192·6	188
Beech-nut	102	112	108-109	191	198	195·5
Cotton-seed	102	111·2	106	191	196·2	193·5
Butter	26	35	33	221	227	224
Cocoa-nut	8	9·35	8·5	253	262	257
Cod-liver	123	166	144-148	175	194	182-187
Earth-nut	87·3	103	94-96	190	197	194
Hemp-seed	140·5	166	150	190	193	191·5
Hazel-nut	88·2	86·9	84·8	191·4	197·1	194
Bone-fat	46	55	49	—	—	190·5
Linseed	170	195	178	187·4	195·2	192
Poppy-seed	134	143·3	138	192·8	194·6	193·7
Walnut	143	152	150	186	197·3	195
Olive	79	88	82-83	185	196	193
Palm	51	52·4	51·5	200	202·5	201·5
Castor	82	85·9	84·5	176	186	180
Beef-tallow	35·5	44	39	193	206	197
Seal	96·5	152·4	128	178	196	192
Rape	98	104	100-101	170	179	177
Sesame	103	112	108-109	187	192	190
Sunflower	122	134	128	189	194	192
Sperm	81·3	84	82·5	117	147	135
Grape-seed	94	99	96 5	178·4	179	178·7
Whale	80·9	130	110	188·5	224·2	192
Japanese wood	149·7	165·7	160	190·7	196·1	194

Wijs' Modification of Determining Iodine Absorption of Oils.—Thirteen grammes of iodine are dissolved in 1 litre of glacial acetic acid on the water bath, and, after cooling, chlorine is passed through the solution until its titration strength is doubled, a rather sharp alteration in the colour indicating the point. The Wijs' solution may also be prepared with iodine bichloride, or with the monochloride, but, however made, it can be got ready in sufficient quantity to last for a long time without fear of its losing strength. In conducting the analysis from 10 to 20 drops of the oil are carefully weighed and put into a wide-mouthed stoppered bottle, pure chloroform or carbon tetrachloride is added, and 25 c.c. of the Wijs' solution containing about 16·24 grammes ICl per litre are next put in. After standing for fifteen minutes potassium iodide solution is further added, and the volume made up to 150 c.c. with water. It is then titrated with thiosulphate in the usual manner.

The Wijs' method agrees somewhat closely with the Hübl, but the former gives rather higher results. Harvey found with rape oil (Hübl's method) 101·6 and 104·9. Cod oil seems to give as much as 7 or 8 per cent. higher by the Wijs' method than by Hübl's. The advantages of the Wijs' process are rapidity (earth-nut absorbs 98·9 per cent. of its full quantity in one minute), stability of solution, and quick and accurate results.

The following table gives Wijs' results with raw linseed oils of different origin :—

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Origin of Oil.	Iodine Value.	Density at 15° C.	Impurities Per Cent.	Foreign Seed Per Cent.
DUTCH OILS :—				
(1) Friesland	201·8	0·9352	—	—
,,	198·3	0·9346	—	—
,,	195·6	0·9333	—	—
,,	195·6	0·9337	—	—
,,	195·3	0·9339	—	—
(2) Groningen	199·3	—	—	—
,,	197·4	—	—	—
(3) Zeeland	193·5	—	—	—
EAST INDIAN :—				
(1) Bombay	187·5	0·9324	—	—
,,	186·0	—	4	—
,,	185·7	0·9320	—	—
,,	185·6	—	4·44	0·3
,,	185·0	—	6·55	0·59
,,	184·7	—	4·86	0·42
,,	183·9	0·9324	4·04	0·28
,,	182·3	0·9313	—	—
(2) Calcutta	182·8	—	4	—
,,	182·2	0·9321	4·14	0·94
LA PLATA OIL	182·7	0·9314	3·01	0·17
,,	180·0	—	2·17	0·60
,,	180·0	—	3·03	0·10
,,	179·6	—	—	—
,,	178·2	—	—	—
,,	177·5	0·9311	—	—
,,	176·3	0·9311	1·78	0·00
,,	174·7	—	4	—
NORTH AMERICA	188·5	—	—	—
,,	182·3	—	2·37	—
,,	178·1	—	5·75	—
,,	178·1	0·9309	5	—
,,	177·8	—	—	—

Origin of Oil.	Iodine Value.	Density.	Impurities Per Cent.	Foreign Seed Per Cent.
NORTH RUSSIA :—				
Archangel	192·1	—	—	—
Wiatka	197·4	—	—	—
,,	196·4	—	5	—
,,	194·0	—	—	—
St. Petersburg	195·0	0·9327	—	—
,,	194·2	—	—	—
,,	188·5	0·9325	—	—
Reval	198·5	—	8	—
Pernau	198·2	—	10	—
,,	196·9	—	5	—
Riga	200·0	—	5	—
,,	195·5	—	20	—
,,	194·2	—	—	—
Liban	195·5	—	5	—
,,	194·6	—	2	—
,,	192·4	0·9335	—	—
MID RUSSIA :—				
Samara	189·1	—	—	—
Steppes	188·9	—	2	—
SOUTH RUSSIA :—				
Azof	182·5	0·9319	—	—
,,	182·1	0·9312	—	—
,,	181·7	—	—	—
,,	181·6	—	—	—
,,	179·9	—	—	—
,,	179·1	0·9314	—	—
,,	178·5	—	4	—
Taganrog	181·7	—	—	—
,,	178·3	—	—	—
,,	177·9	—	8·66	1·29
,,	176·3	0·9305	—	—
Donau	182·1	—	4	—

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15. *McIlheny's Bromine Absorption Method.*—About 0·2 gramme of the linseed oil to be tested is placed in a glass-stoppered bottle, 10 c.c. of carbon tetrachloride added to dissolve the oil, and then 20 c.c. of third normal bromine in carbon tetrachloride run in from a pipette. Another pipetteful is run into another similar bottle. It is convenient, but not absolutely necessary, that both bottles should now be cooled by immersing them in cracked ice. This causes the formation of a partial vacuum in the bottle. The bromine need not be allowed to react with the oil for more than a few minutes as the reaction between them is nearly instantaneous. Twenty-five c.c. of a neutral 10 per cent. solution of potassium iodide is introduced into each bottle by slipping a piece of rubber tubing of suitable size over the lip of the bottle, pouring the iodine solution into the well thus formed, and shifting the stopper slightly so as to allow the solution to be sucked into the bottle, or, if the bottle has not been cooled, to cause the air as it escapes from the interior to be washed by bubbling through the potassium iodide solution. This prevents the loss of any bromine or hydrobromic acid. The iodide solution has been introduced, the bottle is shaken, and set in ice for a couple of minutes more, so that there may be no loss when the stopper is opened, caused by a slight pressure inside the bottle. The solution reaction causes some heat and consequent pressure. The free iodine is now titrated with neutral tenth-normal sodium thiosulphate, using as little starch as possible as indicator. At the end of this titration 5 c.c. of a neutral 2 per cent. solution of potassium iodate and a little more starch solution are added and the iodine liberated, on account of the hydrobromic acid produced in the original action of bromine on the oil, titrated with thiosulphate. From the figures so obtained, the total percentage of bromine which has disappeared is calculated, and the percentage of bromine found as hydrobromic acid, called the " bromine substitution figure," is also calculated, while from these two the " bromine addition figure " is obtained by subtracting twice the bromine substitution figure from the total bromine absorption. If an oil contains rosin, rosin oil, or mineral oil, the fact will be brought out by this process, and an indication given by the figures so obtained as to which one is present. If the bromine substitution figure is normal the absence of more than a very small quantity of turpentine, benzine, rosin, or rosin oil is assured. The process can be carried out in the time necessary for weighing and titrations, as the standard solution, unlike the Hübl solution, does not deteriorate on keeping if tightly closed, so that it is always ready for immediate use, and there is no waiting for some hours for the reagents to act upon the oil, as in the Hübl process, for in this case the reaction takes place immediately.

The bromine *addition* figure of linseed oil lies ordinarily between 100 and 110. Age lowers the halogen figures of linseed oil.

A low " addition figure " may also be caused by the presence of rosin, rosin oil, benzine or mineral oils, which have figures usually

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below 15; by the presence of some other seed oil, the commonest of this class being corn and cotton-seed oils, having figures in the neighbourhood of 73 and 63 respectively; or (see p. 108) by the oil, in case it is a boiled oil, having been boiled in the old-fashioned way at a high temperature. If the "addition figure" is very much higher than 110, it will be found that the oil contains turpentine, as all other foreign materials added have lower figures than linseed oil.

The "bromine substitution figure" of genuine linseed oil is commonly about 3. A much higher figure would point to turpentine, rosin, or rosin oil, which give figures from 20 to 90; to the presence of some petroleum product, as benzine, having a figure in the neighbourhood of 15, or a heavier petroleum oil, which may have as low a figure as linseed, or may be much higher; or to the presence of mineral acid in the oil, which may be allowed for by a separate determination of its amount, as described under the determination of the "acid figure".

It is a noteworthy fact that both the Hübl and the "bromine addition figures" are practically the same for boiled oil as now made as for raw oil, whereas boiled oil made by the old process at a high temperature gave distinctly lower figures on account of the effects of the great heat upon the oil.

The amount of bromine equivalent to the iodine absorbed as expressed by the Hübl figure has been calculated, and by dividing this result by the "bromine addition figure," a figure was obtained for each oil which is intended to express, by the amount it exceeds 1,000, the amount of substitution of iodine which has gone on in the Hübl iodine absorption. For example, if the figure obtained for an oil by the calculation described is found to be 1·075, it indicates that the Hübl figure is in that case 7·5 per cent. higher than the true iodine figure, which should express the iodine absorption by addition.

The "bromine addition figure" is not sensibly affected by the length of time that the oil is allowed to remain in contact with bromine, but the "bromine substitution figure" probably is. The difference between five minutes' and thirty minutes' contact does not appear, however, to be marked, unless the substitution figure is very high, as in the case of pure rosin or turpentine.

In carrying out either the Hübl or the bromine process upon oils it is necessary that an excess of iodine or bromine should be used amounting to as much as the oil absorbs. Many iodine figures on record are too low because this precaution was not attended to.

It is believed that more information is to be obtained as to the character of a sample of linseed oil by determining the bromine figures than by any other single test. In the case of an oil of unknown character, it would in most cases be advisable to apply this test first to it (McIlheny).

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16. The Percentage of Insoluble Bromine Derivatives.

This determination is proposed by Hehner and Mitchell (*Analyst*, Dec., 1898, vol. xxiii., p. 310). It depends upon the fact that linseed oil gives, when dissolved in ether and treated with bromine, compounds of glycerides and bromine which are insoluble in the ether, while oil containing glycerides of oleic acid only, and even semi-drying oils like cotton-seed and corn oils, give soluble compounds. Hehner and Mitchell obtain the following percentages of insoluble bromine compounds from different oils:—

	Per cent.
Linseed oil	23·86 to 25·8
Poppy oil	0·0
Corn oil	0·0
Cotton-seed oil	0·0
Olive oil	0·0
Almond oil	0·0
Rape-seed oil	0·0
Whale oil	25·0
Cod oil	34·5
Cod-liver oil	42·9
Shark oil	22·0

The process, which seems to be a valuable one, detecting adulterations of linseed oil with other seed oils, has been satisfactorily reported on by McIlheny. Two samples of raw linseed, six samples of boiled linseed, two of corn, and one of cotton-seed oil, tested by him, gave results agreeing substantially with those of Hehner and Mitchell. Two samples of mineral oil, one light and one heavy, one sample of rosin oil, and one sample of turpentine failed to give any precipitate of insoluble bromine derivatives.

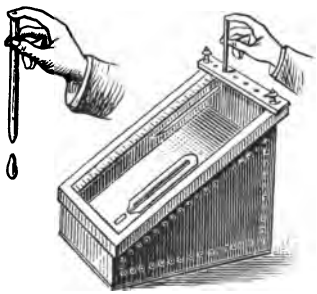

Fig. 29.—Apparatus capable of being Heated for Testing Drying, Viscosity, Gumming, etc., of Oils, with graduated scale to left and thermometer to right.

Rapidity of Drying and Weight of Oxygen Absorbed.

17. Mulder pointed out the necessity of spreading the oils out thinly so that they did not dry on the surface and thus prevent the liquid underlayer from absorbing oxygen. He spread 3 grammes of

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oil over 220 square centimetres so as to get about 0·015 gramme of oil per square centimetre. Under these conditions he noted the following increases:—

Oil.	Increase in Weight Per Cent.	Oil.	Increase in Weight Per Cent.
Poppy-seed	12·2	Linseed and Manganese Borate	12·4
Walnut	8·7	Linseed and Litharge	12·5
Linseed	11·9	Linseed and Red Lead	13·2

Casselman tried to lessen the time required by the oil to absorb its maximum of oxygen by heating 3 to 4 grammes of the oil for three hours daily up to 150° C. (302° F.), with the following results:—

Oil.	Behaviour after above Treatment.
Linseed	Dry after 1½ to 2 days
Poppy-seed	,, 4 to 5 ,,
Hemp-seed	,, after a few days more
Sunflower	Viscid and gelatinous after 3 months

But this method is altogether too irregular for serious consideration.

Kissling spread 10 grammes of several oils over 35 square centimetres and exposed the same for ten days at the ordinary temperature, with the following results:—

Oil.	Increase in Weight Per Cent. at Ordinary Temperature.	Oil.	Increase in Weight Per Cent. at Ordinary Temperature.
Olive	0·0	Cotton-seed	0·545
Rape (crude)	0·05	Raw Linseed	1·130
Rape (refined)	0·000	Boiled Linseed	3·400
Neatsfoot (refined)	0·065	Triolein	0·0

Livache's Precipitated Lead Test.—The transformation of a drying oil into a solid elastic substance is due to an absorption of oxygen. Linseed oil absorbs 14 to 16 per cent. of its weight, and it is easy to directly measure the quantity absorbed, by imbibing in the oil a little spongy metallic lead well washed and dried *in vacuo*, obtained by

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precipitating a lead salt by a zinc plate; the whole is exposed to air as long as the weight varies; the increase in weight indicates the amount of oxygen absorbed.

By this process, when conducted with the necessary precautions, the quantity of oxygen absorbed by any sample of linseed oil may be rapidly ascertained, and, whether a pure oil is being dealt with or an adulterated oil, or an oil already partially oxidised, in all cases it can readily be ascertained whether an oil corresponds with a given sample.

This reaction ought to draw the attention of practical men, because samples are often met with which, although equally genuine, take longer time to dry. Now, the precipitated lead test indicates the difference in duration of the time taken by the oil for complete oxidation, and thus predicts beforehand the way in which any given oil will dry when applied to an appropriate surface on the large scale.

Process.—One gramme or thereabouts of the precipitated lead is weighed in a tared watch glass, then 0·5 gramme of oil is spotted upon it drop by drop, spacing out the drops in such a way that a little dry lead rests between each of them. At the end of two days the following results were noted in regard to oils in column I. The oils in column II. showed no results during the first two days. The figures in the column are the increase after a week.

I. Oil.	Increase Per Cent.	II. Oil.	Increase Per Cent.
Linseed	14·3	Colza	2·9
Walnut	7·9	Sesamum	2·4
Poppy-seed	6·8	Earth-nut	1·8
Cotton-seed	5·9	Rape	2·9
Beech-nut	4·3	Olive	1·7

Jean has tested Livache's method with fish oils in dry air under a bell jar with H_2SO_4, with the following results :—

Oil.	Increase in Weight Per Cent.	Oil.	Increase in Weight Per Cent.
Whale	8·26	Menhaden	5·45
Japanese Fish	8·19	Sperm	1·63
Cod-liver	6·38		

Weger tested Livache's method against the same oil on a glass slab by itself, and also on a glass slab with both red lead and litharge as driers respectively. He found that the amount of lead in Livache's method must be increased in order to get a sharp maximum.

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Livache's Method. Increase in Weight.		The same Oil spread on a Glass Slab.	The same Oil with 6·6 Grammes of Red Lead to 0·3188 Grammes of Linseed Oil.		The same Oil with 10·4 Grammes of Litharge to 0·4910 Grammes of Linseed Oil.	
Days.	Increase Per Cent.		Days.	Increase Per Cent.	Days.	Increase Per Cent.
1	11·4	Gave a	1	2·10	1	2·00
2	12·2					
3	12·4	Maximum of	2	11·30	2	11·30
4	12·6					
5	—	17·1	3	14·14	3	13·64
6	12·9					
7	—	on a	4	14·65	4	14·32
8	13·5					
15	14·8	Glass Slab in	5	14·59	5	14·27
42	17·2					
53	18·1	5 to 6 Days.	7	14·05	7	14·21
85	20·4					

Weger found the following oxygen absorption addition figures :—

Oil.	Days.	Maximum Increase Per Cent.	Oil.	Days.	Maximum Increase Per Cent.
Rape-seed - -	7	7·6[1]	Electro Boiled -	1½-2	15·1-16·7
Olive - - -	20	5·2[1]	Commercial Boiled		
Pine-seed - -	29	10·5[1]	(Hydrated Oxide		
Palm-kernel - -	13	0·8[1]	of Manganese) -	1·2	14·7-14·8
Air-blown Rape -	15	7·7[1]	Commercial Boiled		
Hemp - - -	4-4½	13·4-13·6	(Litharge) - -	16-24 hours	14·61-14·8
Poppy-seed - -	6½	13·4			
Wood (Various) -	3-9	13·4-15·9	Boiled Linseed prepared with 3 per cent. of Rosinate of Manganese in the cold - -	24 hrs.	14·7-16
Indian Linseed -	4½-6	16·8-17·3			
Commercial Linseed - - -	4½-8	17-18·7			
English Linseed -	3½	19·7-19·9			
Old Tanked Linseed	3	15·1-15·7			
Indian Linseed (heated for a short time at 150° C.) -	6	17·0	Boiled Linseed prepared with 3 per cent. of Lead Manganese Rosinate in the cold	—	17·2
Indian Linseed Blown (cold) -	8	16·7			
Indian Linseed Blown (hot) -	5½-6½	8·2-9·3	The same after 18 months' exposure to the air - -	—	10·9
Stand - - -	18	11·1			
Thickened - -	18	10·7	Boiled Rosin oil -	7½	23·3

Bishop's Silica and Rosinate of Manganese Test.—1. Ten grammes of the oil are weighed in a capsule, and 2 grammes exactly of the rosinate of manganese added. The capsule is put into the water

[1] Oxygen absorption addition still proceeding.

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bath, stirring it from time to time until the complete solution of the rosinate, from five to ten minutes being sufficient for this. It is then cooled.

2. One gramme of silica is weighed in a capsule with a flat bottom furnished with a little glass stirrer. With the aid of a dropping tube, a quantity of oil, as near as possible approaching 1·02 grammes (*i.e.*, 1 gramme of oil plus 0·02 of rosinate), is allowed to fall drop by drop over the whole of the surface. Both the weight of the oil and the total weight are noted. By means of the stirrer the oil is mixed with the silica in such a way as to have a divided mass, perfectly homogeneous, covering the whole of the bottom of the capsule. This is left at a temperature of from 17° to 25° C. in the case of drying oils, of 20° to 30° C. for the others, and weighed at the end of varying periods; for instance, six hours, sixteen hours, twenty-two hours—that is to say, three times in the twenty-four hours. After each weighing the surface was renewed by agitation with the stirrer. The degree of oxidation is furnished by the maximum augmentation multiplied by 100 when 1·02 grammes weighed exactly is operated on. Using pure linseed oil and the same oil with 2 and 4 per cent. of rosinate the following were the results. The linseed oil had a specific gravity of 0·9322 at 15° C., and through the experiments the temperature ranged from 20° to 25° C.:—

Increase per cent. at the end of	No Drier added.	2 per cent. of Rosinate added.	4 per cent. of Rosinate added.
6 hours	0	12·35	11·10
22 ,,	0·50	15·65	15·50
24 ,,	0·80	15·85	15·30
30 ,,	2·50	16·25	15·30
48 ,,	7·30	15·65	14·90
72 ,,	15·00	14·65	14·10
96 ,,	16·40	15·15	13·60
120 ,,	15·30	14·05	13·20
144 ,,	14·90	13·75	13·20
168 ,,	14·30	13·35	13·20
288 ,,	14·00	13·25	13·10

These experiments showed in a decisive fashion the energetic action of rosinate of manganese as a drier, since this salt produced the total oxidation of the oil three or four times as quickly as in operating with simply divided oil. It also confirms the conclusions of the experiments of Cloez and those more recently made by Mulder, Bauer, Hazura and Livache on the formation not only of solid products but also of volatile products. If the oxidation is too strong, as in the case where 4 per cent. of rosinate was added, a final result is obtained very much more quickly and at the same time a feebler increase, this phenomenon being explained by reason of the formation

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of a higher proportion of volatile products. In the experiments that follow, therefore, 2 per cent. of rosinate has been used:—

Increase per cent. at the end of	Pure Linseed Oil. Sp. gr. 0·9322. Temperature 17° to 23° C.	Linseed Oil containing 5 per cent. of Rosin Oil and 5 per cent. of Mineral Oil. Sp. gr. 0·9323. Temperature 17° to 23° C.
6 hours	13·50	11·50
22 ,,	16·30	14·80
24 ,,	16·40	14·90
30 ,,	16·20	14·80
48 ,,	15·90	14·60
120 ,,	14·80	13·80

The influence of temperature is very great, and it would seem useful in the case of linseed oils to operate at a temperature not exceeding 28° C., but above 17° C., in order to effect the oxidation in as short a time as possible. The experiments showed also the inferiority of exotic linseed oils of a low specific gravity, and also showed that it is possible to establish by a comparison the inferior quality arising from clever adulteration.

Oils.	Specific Gravity.	Average degree of maximum oxidation.	Ratio $\frac{17·05}{x}$
French Linseed	0·9327	17·05	—
Linseed from La Plata	0·9304	15·20	1·12
Hemp-seed	0·9287	14·40	1·18
French poppy-seed	0·924	14·20	1·20
Commercial walnut	0·924	19·70	1·23
Demargarinated cotton-seed	0·923	3·45	1·80
Non-demargarinated cotton-seed	0·924	8·60	1·98
Senegal sesamum	0·9215	8·70	1·96
Indian sesamum	0·921	7·40	2·30
African earth-nut	0·916	6·70	2·54
White earth-nut	0·916	6·50	2·62
French colza	0·9142	6·40	2·66
Indian colza	0·9137	5·85	2·91
Olive	0·9155	5·30	3·21

The degree of oxidation can therefore be advantageously used to control the iodine value, and in many circumstances it is even capable of replacing it with advantage. In fact, in addition to its great simplicity and the minimum cost of carrying it out, which makes it a very practical method for the industrial laboratory, *this method furnishes very often a more rational and more complete indication than the Hübl number*, as it permits many minor interesting details to be noticed. In consequence, this process will serve to fix the value and establish the nature of the identity of an oil, whether taken separately or compared with a type. Moreover, it may be applied in the case

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of certain mixtures, not only of oils among themselves, but also of commercial products, such as lard and alimentary fats, which often contain very variable quantities of vegetable oils. In this last case, instead of oxidising directly either the melted and clarified fat or the fluid part extracted by pressure, the liquid fatty acids may be operated upon, separated from the solid acids by Halphen's process. The oil is saponified, the soap dissolved in water, and precipitated with acetate of lead. The lead soap is extracted with ether, and the ethereal solution of the lead salts of the non-saturated fatty acids is precipitated with weak sulphuric acid. Indications still more interesting and more precise are obtained by taking the iodine value before and after oxidation. The determination of the degree of oxidation gives results in immediate connection with the value of the oils for certain of their industrial purposes, and, as has been shown, certain falsifications can be detected by the process. Moreover, it will show whether recently crushed or old tanked oil is being dealt with.

18. *Maumené's Test.*—1. The sulphuric acid to be used in this experiment should be pure, of specific gravity 1·845, and should be kept in a well-stoppered and capped bottle. The stopper should not be left out a moment longer than that required to extract the necessary quantity. Have the acid in a bottle 6 inches high, with a thermometer inside.

2. Counterbalance a glass tube, on foot, standing heat (about 1½ inches in diameter and 7 ounces capacity), and weigh in accurately 50 grammes of the oil.

3. Immerse both the sulphuric acid and the tube containing the oil in water contained in a tin vessel 5 inches deep, 2 quarts capacity, and apply heat. As soon as both the acid and the oil are at a temperature of 27° C., draw out 10 c.c. of the acid with a pipette and let it flow gradually at the rate of 1 c.c. every five seconds into the oil, without touching the sides, stirring very energetically all the time with the thermometer. After all the acid is in continue to stir exactly for half a minute, then move the thermometer more slowly, noting the exact degree at which it ceases to rise.

A pure sample of oil should be tested in the same manner precisely, immediately before the suspected article. No two persons, unless actually working side by side, will ever get absolutely identical figures, and rarely then. So delicate is the rise of temperature that the same operator must needs be careful to obtain constant figures on repeating the experiment on the same oil.

In the case of linseed oil and fish oils the action is very energetic, abundant fumes being given off, whilst the mixture, assuming a gluey consistence, thorough mixing of the acid and oil is a matter of great difficulty. Moreover, it is not at all easy to obtain satisfactory readings of the thermometer, the more so as the latter has to be used to stir the oil. It is therefore advisable to mix linseed, etc., oils in known proportion with an oil on which sulphuric acid has no great action, such as petroleum lubricating oils.

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PERCENTAGE OF OXYGEN ABSORBED BY DIFFERENT STRIPS OF CHAMOIS EXPOSED TO THE AIR AND IMPREGNATED WITH DIFFERENT OILS AND COMPARED AGAINST A BLANK STRIP.

	Iodine Value.	Absorption of Oxygen in 100 parts of Oil after														Maximum.	Absorption according to Livache.	Calculated from the Iodine Value 1 × 0·063
		1 Day.	2 Days.	3 Days.	4 Days.	5 Days.	6 Days.	7 Days.	8 Days.	9 Days.	10 Days.	14 Days.	21 Days.	28 Days.	56 Days.			
Blank Test -	—	0·0	1·0	1·3	0·9	1·4	−0·6	1·3	0·0	0·5	1·8	−0·5	1·1	3·3	−2·5	—	—	—
Olive Oil - -	82·1	0·2	1·0	1·2	0·9	1·4	−0·4	1·1	−0·1	0·5	1·7	−0·5	0·7	3·5	−2·5	0·2	1·7	5·2
Sesame Oil -	110·2	0·1	1·2	0·9	0·5	1·1	−0·5	1·0	0·0	0·7	2·0	1·1	4·7	6·6	−0·5	3·6	2·4	7·0
Rape Oil - -	102·4	0·1	1·1	1·3	1·0	1·8	0·5	2·5	2·0	2·8	4·6	2·3	3·2	6·3	−0·2	2·8	2·9	6·5
Cotton-seed Oil	109·2	−0·1	0·8	1·4	1·2	3·1	2·2	5·0	4·7	5·7	7·4	2·5	1·6	5·2	−1·4	5·6	5·9	6·9
Poppy-seed Oil	135·9	0·3	2·0	3·2	4·3	7·1	7·3	9·7	7·0	7·3	8·0	3·3	2·7	6·7	−0·3	8·4	6·8	8·6
Walnut Oil -	149·2	−0·2	2·0	4·4	7·1	9·7	8·4	9·6	7·2	7·3	8·3	4·0	4·2	8·4	−1·3	9·0	7·9	9·4
Linseed Oil -	175·8	0·1	1·5	2·0	3·8	12·3	11·8	13·2	10·4	11·3	11·8	7·9	8·2	12·6	4·5	12·4	14·3	11·1
Cod-liver Oil -	171·0	−0·6	1·1	8·1	10·0	10·9	8·0	10·4	8·0	8·5	10·1	6·8	6·3	11·3	4·3	9·5	6·4	10·8

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Knowing the heat given out by mixing the mineral oil alone with sulphuric acid, it is easy to calculate how far its admixture with linseed oil has affected the latter.

MAUMENÉ'S TEST, RESULTS WITH TYPICAL OILS.

Oil.	Maumené.	Baynes.	Archbutt.	Allen.	De Negri and Fabris.
Linseed	103	104-124	—	104-111	122-126
Hemp-seed	98	—	—	—	95-99
Walnut	101	—	—	—	96
Poppy-seed	74	—	86-88	—	87-88·5
Niger-seed	—	82	—	81	—
Sunflower	—	—	—	—	72-75
Soja-bean	—	—	—	—	117
Cotton-seed (crude)	—	84	70	67-69	—
,, (refined)	—	77	75-76	74-75	—
Olive	42	40	41-45	41-43	32-37
Dolphin	—	—	42	41-47	—
Menhaden	—	—	123-128	126	—
Cod-liver	102-103	116	—	113	—
Whale (Arctic)	—	—	—	91	—
,, Antarctic	—	—	92	—	—
Seal	—	—	—	92	—

Specific Temperature Reaction (Thomson and Ballantyne).—The rise of temperature obtained by mixing 50 grammes of oil with 10 c.c. of sulphuric acid, divided by the rise of temperature produced by mixing 50 grammes of water with 10 c.c. of the same sulphuric acid, in the same vessel and under identical conditions, gives a quotient termed the specific temperature reaction.

Origin of Oil.	Authority	Specific Temperature Reaction.	Origin of Oil.	Authority.	Specific Temperature Reaction.
Baltic	Thomson and Ballantyne	349	River Plate	Thomson and Ballantyne	320
East Indian	,,	320	—	Jenkins	313

19. *Elaidin Test for Differentiating between Drying and Non-drying Oils.*—This is a very simple test, requiring no costly apparatus, when applied according to Poutet's original instructions. The proper way to apply the test is as follows:—

A. Apparatus required:—

1. Supply of 2-ounce conical test glasses on feet.
2. A swan-neck minim measure.
3. Mercury ¼ lb. in narrow-mouthed stoppered bottle.

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4. Glass rods rounded at one end and dubbed out at the other.
5. Glass flask, 30 ounces.

B. Preparation of the test solution: Weigh out in any suitable glass vessel, such as a small beaker, 60 grammes of metallic mercury, run it into the capacious flask; place the latter in a vessel of water in a stink-closet, or in a good draught; then add carefully and gradually, a little at a time, 75 grammes of nitric acid, specific gravity 1·37 (74° Twaddell). Keep the flask well immersed in the water to condense the fumes, which are partially retained by the solution and constitute its acting principle. Transfer to a well-stoppered bottle, and label it olive oil test with date, as it does not keep long.

Process.—Pour into a 1-ounce conical test glass on foot 10 minims of the above solution. From a separate minim measure run in 100 minims of oil on to the top of the mercury solution. Thoroughly incorporate the mercury solution and the oil by stirring continuously with a small glass rod, dubbed out at the end by having been heated till viscous in a bunsen or blowpipe flame, and then pressing it against a slab of slate, stone, etc. Repeat the test on a known sample of pure olive oil, and with olive oil adulterated, respectively, with 5 per cent., 10 per cent., 15 per cent., 20 per cent., 25 per cent., and so on of any drying oil. The test is best performed at five o'clock at night, and the set of samples set aside until next morning at nine with their respective rods left in the glasses.

Treated in this manner olive oil gives a canary-coloured hard mass which can be lifted completely out of the test glass by means of the glass rod, and while still adhering thereto if it be struck against the test glass it emits a metallic resonant sound or musical note.

Most writers refer only to the time which the oils take to solidify under the action of this reagent. We prefer, as the result of prolonged experience in the testing of olive oil, to take the ultimate hardness and colour after a reasonable time, say, 5 P.M. to 9 A.M., as the standard to go by, and our results have never been called in question. It would be well if innovating analysts would test the original process thoroughly before modifying it for the mere sake of the vainglory of adding one more useless modification to numerous others.

Lallemantia Oil.

French, *Huile de Lallemantia;* German, *Lallemantiaöl.*—Density at 20-21° C., 0·9336; solidifies at − 35° C.; Hehner value, 93·3; Reichert value, 1·55; saponification value, 185; iodine value, 162·1; melting-point of fatty acids, 22·2° C.; solidification point of fatty acids, 11° C.; iodine value of fatty acids, 166·7. This oil is extracted from the seeds of *Lallemantia Iberica, Fisch et M.*, a plant belonging to the *Labiatæ*, growing wild in the Caucasus, and cultivated as a farm crop in Russia, near Kieff. Its iodine value is very high, thus ranking it amongst the first

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members of the drying oils. Wood oil, however, surpasses it, although the drying properties of linseed oil would appear, according to Richter, to be inferior to it. Richter spread a little of the oil on a watch-glass, and after nine days' exposure it dried to a thick resinous coating. But the drying properties have possibly been overrated, and confirmatory results are necessary. By heating the oil to 150° C. (302° F.) for three hours complete drying took place after twenty-four hours. The oxygen absorption by Livache's test was 15·8 per cent. after twenty-four hours, and for the mixed fatty acids 14 per cent. after eight days.

Ten grammes of the oil at 18° C. treated with 2 grammes of concentrated sulphuric acid gave an increase in temperature of 120° C. (248° F.).

Wood Oil.[1]

Wood oil, the best drying oil known, is produced by the oil tree of China and Cochin China, the *Elæococca vernicia* (Tong Yeou)—a plant belonging to the *Eurphorbiaceæ*, or spurge worts, which flourishes on the banks of the Yanktse River in China—the seeds of which contain 53 per cent. of oil, 80 per cent. of which is got by crushing. In China it is used to some extent as an illuminant, but its consumption is chiefly as a varnish for boats, houses and furniture. It is also largely used in the varnishing of the native umbrella, and in the manufacture of waterproof cloth, whilst it is practically a necessity in the manufacture of the finest silks. Of late years it has even been used in the building of forts, forming with lime, sand and clay a material almost as tough as granite. In the western provinces the oil is adulterated in order to lessen its cost and to bring it into consumption for "coarser" purposes. Owing to the distance which the product has to be brought to export ship, the great cost of repacking (an absolute necessity) and freight, its lay-down price is heavy.

Until lately the only literature on wood oil consisted of short papers by Davies and Cloez, but a large number of workers have recently busied themselves with this product, such as Holmes, De Negri and Sburlatti, Jenkins, Williams, Fraps, Jean, Kitt and Milliau. The crushing of wood-oil nuts has unfortunately not yet been done on a manufacturing scale in Europe, as there seems to be some doubt both of the regularity of the supply and of the value of the press-cake.

There are no reliable statistics of the Chinese wood-oil nut harvest. In 1899 Hankow and other Chinese ports exported about 30,000 tons of wood oil, and it is considered that China herself uses double that quantity. This would make the total output of oil about 90,000 tons, and of nuts about 160,000 tons. The nuts themselves are not exported. It seems fairly certain, however, that a real demand would

[1] Cloez published the results of a careful study of this oil in the *Comptes Rendus de l'Académie de Science*, vols. lxxxi., lxxxii. and lxxxiii. It must not be confounded with gurjun balsam, a hydrocarbide oil also put on the market as wood oil.

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soon be followed by an increase in the cultivation of the nut. The well-known variability in the quality of wood oil is due on the one hand to its coming from different species of the tree (*Aleurites cordata*), and which are as yet imperfectly known, and on the other hand to the very primitive manner in which the Chinese extract the oil. The exporters at Hankow recognise three sorts: pai-yu, hsin-yu and hung-yu, but do not themselves know whether the differences are due to different sources of origin or to one being a first, another a second pressing, or one being hot pressed, and another cold pressed, or other differences having existed in their treatment. The investigations, the results of which are given below, were carried out with a small quantity of nuts, of which the exact locality of origin is unknown, except that it is Chinese. A slightly modified form of arachis-sheller was found to extract the kernel perfectly, and the kernels were 52 per cent. of the weight of the whole nut. The shells are of no value, and this is a matter of great importance, for it is evident that freight would be saved if the nuts were shelled in China. It is true that the kernels are very prone to become rancid when deprived of the protection of their shell, but they would not suffer during the voyage if packed air-tight. The kernels gave 58·7 per cent. of oil by extraction with ether. A pressure of 350 atmospheres on the other hand, at 28° C., only gave 43·0 per cent. The residue from this pressing, when broken off and again subjected (between rollers) to a pressure of 350 atmospheres at 65° C., gave 10·7 per cent. of the 15·7 per cent. still remaining. Thus the total yield was 53·7 out of a possible 58·7 per cent.

Hence the yield reckoned on the whole nut is 22·36 per cent. on the first pressing, and 5·56 per cent. on the second, with 24·08 per cent. of oil-cake. The oil from the first pressing was a pale yellow, and that of the second a pale orange, and more viscid than the first. The constants of both oils agreed substantially with those already obtained by other investigators. It was found, however, that by picking the nuts before pressing and rejecting any that showed any sign of rottenness, the acid number of the oil of the first pressing could be brought down to 0·9. The oil has also no longer the disagreeable characteristic wood oil odour, and can be kept for weeks in a well-stoppered bottle without developing it. If exposed to the air, however, it quickly acquires it. This seems to confirm Ulzer's opinion that the development of the smell is a result of oxidation. But possibly the oil could easily pass through a sea voyage, in well-filled and closely stoppered vessels, without acquiring any of the disagreeable odour. The press-cakes are very poisonous, and contain 53 per cent. of protein and 12·1 per cent. of fat. The Chinese use them for manure, and also burn them for the manufacture of lamp-black. If some means could be discovered of extracting their poisonous ingredients the high percentage of protein which they contain would make them an excellent fodder.

Properties.—The oil, when of good quality, is somewhat viscous,

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pale amber in colour, rather dull, lacking that brightness which is seen in rape or cotton-seed oil. It has a peculiar and characteristic nutty odour and unpleasant taste.

Solubility.—Chinese wood oil is soluble in ether, petroleum ether, chloroform, and also in amyl alcohol, but is insoluble in cold absolute alcohol.

Drying Properties.—A coat of wood oil on a slab of glass dries completely in a few hours at the ordinary temperature. Light alone, without air, is said to cause it to dry. When heated to 200° C. it thickens; at 260° to 280° C. the oil gelatinises, but the jelly does not stick to the fingers. It should therefore not be heated above 180° C. Unlike other drying oils it does not dry from without inwards, but the process goes on simultaneously through the thickness of the coat of oil. This oil is well adapted for mixing with linseed oil, the latter component giving elasticity and the former hardness and resistance, as well as an increase in drying properties.

By reason of its great siccative power elæococca oil can be successfully employed instead of linseed oil in making copal varnishes, and makes them very durable and elastic. It requires no boiling to make it drying, but when boiled with oxide of lead and dissolved in turps it makes a fine varnish for furniture without any resin. It can also be used for making paper and cardboard impermeable. Paper prepared with it is transparent, and can be written on with ordinary ink. Another use of elæococca oil is to replace linseed oil in the manufacture of oil colours, thereby escaping the darkening effect of the latter oil.

The attention of varnish manufacturers is directed to this oil. It is a known fact (says Livache) that large quantities are yearly imported into England, but for what purpose is unknown. Now its remarkable drying capabilities in the cold, and without previous heating, suggest to him its direct use in England in the manufacture of high-class varnishes. This assertion has recently been reiterated by Ferdinand Jean.

But against its having been used to any extent in England, at least until recently, is the fact that the British oil merchants, on the other hand, advertised it as being *extensively used on the continent.*

The results of experiments with this oil are very conflicting; none of them, moreover, seems to have been undertaken in a truly scientific spirit. Instead of trying to find out the cause of the gelatinisation of the oil on heating, unscientific attempts on rule-of-thumb principles have been made to make concoctions from it somehow or other, so that it may dry after the manner of linseed oil. The examination of this oil will well repay thorough investigation, and possibly it may be found to contain a principle analogous to bird-lime or the serum of india-rubber. In any case, all efforts should be concentrated, in the first instance, to the isolation of the gelatinous principle and the examination of its properties.

In heating wood oil for the purpose of making varnish it must

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always be borne in mind that the temperature must never exceed 180° C., as the oil then gelatinises and will not mix with the other ingredients of the varnish. But with care the result is invariably extremely good. If the oil be heated with a drier and then thinned with French oil of turpentine the result is always good, although prolonged stirring is necessary. Experiments have shown that borate of manganese is not a suitable drier for wood oil, but borax answered extremely well. Mixtures of wood oil and linseed oil were also tried; the varnish dried in twenty-four hours on glass, and after a little became perfectly hard and transparent. But when the dried varnish was exposed to the weather, damp, dew and rain loosened its adhesion —which was, however, fully restored by exposure to dry air or sunlight. On wood the varnish resisted very much better, especially when mixed with zinc white.

The following are typical experiments made on wood oil:—

(*a*) Fifteen pounds of wood oil having been warmed up, 5 lb. of acetate of lead were added to it, a little at a time, the heat being kept up until solution was complete. Ten pounds of gum resin were then added, and when this was melted the varnish was thinned with 10 lb. of French turpentine oil. The varnish so prepared dries in ten hours. A coating of it on tinplate, which was then steeped in a solution of sodium carbonate for twenty-four hours, was not affected except as regards its colour. The latter was made somewhat paler, but the adhesiveness of the varnish was not diminished. Varnishes made with linseed oil and subjected to the same treatment were practically destroyed by the alkaline solution, and could be afterwards removed without difficulty.

(*b*) Ten pounds of rosin were melted up with 5 lb. of borax. On to this mixture 15 lb. of wood oil were poured. The whole was then heated up and mixed with 20 lb. of French turpentine oil. The varnish thus obtained dried rapidly for an hour, but as soon as the turpentine had all disappeared the rate of drying became very much slower, and after fifteen hours the varnish was still sticky. This seems to indicate a good varnish for gilding. The varnish was completely dry in forty-eight hours.

(*c*) This experiment resulted in a very good varnish, which, however, requires very great care in its preparation. Fifteen pounds of wood oil were mixed with 2 lb. of gum resin and driers, and the mixture was heated to frothing. Then 10 lb. of rosin were added, and when the latter was melted the heating was continued to as high a temperature as possible without gelatinising the wood oil, careful stirring being kept up until the liquid began to seem stiff. The vessel was then removed from the fire, and 40 lb. of French turpentine oil were carefully and thoroughly stirred into its contents. The varnish so prepared looks a bit turbid at first, but becomes quite clear within fourteen days. Spread on glass it dries to a hard and elastic coat in eight hours, or ten at the most, and on wood it dried in five hours. When applied to glass it had a fine lustre, and was

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entirely transparent. Any resin can be used for this varnish provided its melting-point is below 180°, as is, for example, the case with soft manilla copal.

(*d*) The varnish made by this process would suit admirably as a thinning medium for oil colours. The ingredients were 15 lb. of wood oil, 15 lb. of linseed oil, and 4 lb. of rosin. After the usual heating the varnish is thinned with 20 lb. of French turpentine oil. The varnish so prepared dries within twenty-four hours. As above mentioned, its adhesion is destroyed by damp. In spite of containing linseed oil it always retains a whitish tinge.

The odour of the Chinese wood oil is a very peculiar one and adheres to the dry coating made with it; it adheres so strongly that it is plainly noticeable even after oilcloth-like goods dried in hot air have been lying for months, the same as oilcloth will always, even after years, smell of linseed oil. Naturally, this peculiar lard-like odour also shows itself with the varnishes produced from the oil, and it therefore becomes necessary to remove it by some process.

In the manufacture of leather and waxcloth an odourless and cheap varnish, which will dry very quickly, is a prime desideratum. Until lately dry linseed oil was the favourite substance used. Of late, however, wood oil has been tried, and gave highly satisfactory results in respect of drying qualities, but had the great drawback of possessing a very disagreeable smell. The following four methods have been tried with a view of removing this only obstacle to its replacement of drying oil which is considerably dearer:—

1. Extraction of the odorous bodies by solvents.
2. Extraction of the odorous bodies (which are possibly aldehydes and ketones) by sodium bisulphite.
3. Treatment with substances at the same time bleaching and deodorising.
4. Treatment with superheated steam.

We proceed to treat of these four methods *seriatim*.

1. The solvent principally tried was alcohol, in strengths of 20 and 40 per cent. When the oil had separated out after having been shaken up with the spirit, it was dried in a stream of warm carbonic acid. It is true that the disagreeable smell was diminished, but the results were unsatisfactory, even without regard to the cost of the method.

2. This method was tried by boiling the oil for six hours with twice its volume of a 10° B. solution of sodium bisulphite with a reflux condenser. The oil was then washed with water from the excess of bisulphite. Its evil odour was not then appreciably less. This shows clearly that the malodorous substances present are neither ketones nor aldehydes.

3. Animal charcoal, silicate of magnesium and the residues of the manufacture of potassium ferrocyanide were tried, without success.

4. By this method very interesting results were got. The first trial consisted in blowing steam at 130° C. through the wood oil for

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eight hours. The nasty smell was perceptibly lessened, but the oil partly solidified on cooling. When the steam was used at 165° C. a marked diminution of the bad odour took place, even in two hours, and in five there was very little smell of any kind, what there was being much like that of linseed oil. In twelve hours the oil had begun to deposit and had regained some of the disagreeable odour. This recrudescence of the smell was, however, only slight when the oil was cooled without contact with air, showing that the action of atmospheric oxygen has probably something to do with the formation of the evil-smelling substances.

Attempts to conceal the bad odour were made, especially with birch-tar oil, but were quite unsuccessful.

CHEMICAL AND PHYSICAL PROPERTIES OF WOOD OIL.

(*Refractive Index*, 1·503.)

Observer.	Density at 15° C.	Solidification Point.	Hehner's Value.	Saponification Value.	Iodine.
Davies and Holmes	0·940	—	—	211	—
De Negri and Shurlate	0·936	—	—	156-172	159-161
Jenkins	0·9343-0·9385	below – 17° C.	96-96·4	194	149·7-165·7
Ulzer	—	—	—	194	162
Williams	0·9415-0·9432	—	96·3-96·6	190·7-196·1	155·4-165·6

FATTY ACIDS.

Observer.	Melting-Point Degrees C.	Solidification Point Degrees C.	Iodine Value.	Heat of Bromination.
De Negri and Shurlate	43·8	31·2	159·4	—
Jenkins	30-37	34·0	—	21-22·1
Williams	40-49·4	37·1	144·1-150·1	—

Hemp-seed Oil.

Hemp-seed oil is expressed from the seed of the hemp plant (*Cannabis sativa*), a plant belonging to the family of *Urticaceæ*, and from the fibres of which cordage, ropes, etc., are spun. Hemp is extensively cultivated in France, Russia, India, etc. Freshly expressed oil is greenish yellow; however, after a time the green tint disappears, the oil assuming a brownish-yellow colour. It has a powerful disagreeable odour of hemp and a bitter taste. The oil does not dry so well as linseed oil, which it is used to adulterate. It finds a legitimate use in the manufacture of soft soap.

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CHEMICAL AND PHYSICAL PROPERTIES OF HEMP-SEED OIL.

	Density at 15° C.	Solidification Point.	Sapon fication Value.	Iodine Value.	Maumené's Test	Oleorefractometer.
Allen - - -	0·925-0·931	Thick	—	—	—	—
Benedikt - - -	—	at	—	157·5	—	—
Château - - -	0·9270	−15° C.	—	—	—	—
De Negri and Fabris	0·9280	solidifies	192·8	140·5	95-99	—
Fontenelle - -	0·9276	at −27° C.	—	—	—	—
Hübl - - -	—	—	—	143	—	—
Jean - - -	—	—	—	—	—	+30
Massie - - -	0·9255	—	—	—	—	—
Maumené - -	—	—	—	—	98	—
Shukoff - - -	—	—	192-194·9	157-166	—	—
Souchere - - -	0·9255	—	—	—	—	—
Valenta - - -	—	—	193·1	—	—	—

FATTY ACIDS.

	Melting-Point Degrees C.	Solidification Point Degrees C.	Mean Molecular Weight.	Iodine Value.	Acetyl Value.
Benedikt and Ulzer -	—	—	280·5	—	7·5
De Negri and Fabris	17-19	14-16	—	141	—
Morawski & Demaki	—	—	—	122-125·2	—
Hübl - - -	19	15	—	—	—

Poppy-seed Oil.

Poppy-seed oil, so highly esteemed by artists, is obtained by pressing the seeds of the poppy (*Papaver somniferum*), indigenous to the Eastern Mediterranean countries, but cultivated generally all over the world, partly for opium, partly for the oil in the seeds. There are two varieties of the poppy: *Papaver album, D.C.*, with white seeds, and *Papaver nigrum, D.C.*, with black seeds. The white poppy yields the best oil, and is the seed used for medicinal purposes. The black poppy is the variety most extensively cultivated, as an oil producer being the most profitable. The yield of fatty oil is about the same in both varieties, *viz.*, about 60 per cent.

According to Sacc the poppy seed contains:—

Oil	54·61 per cent.
Protein	23·26 ,,
Albuminoids	12 ,,
Cellulose	6 ,,
Ash	2 to 3 ,,

Poppy seed does not contain morphine as alleged.

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Extraction.—The poppy heads are opened after being partially dried, and their contents discharged on to an iron plate; they are then winnowed to remove fragments of the capsule, and afterwards ground to meal, which is packed in bags made of canvas and pressed, the oil being caught in tubs, where it is left to settle and thoroughly clarify. Two kinds of oil are on the French market. The white salad oil, the oil from the first pressing of the best quality seeds. This oil is almost colourless, or of a very pale golden yellow. The second quality, used for technical purposes, is the result of the second pressing at a higher temperature and is much inferior. Sometimes it is the result of pressing inferior seed. The white, freshly extracted oil is inodorous with a bland flavour; it is thus largely used as salad oil. Unsound oil is acrid.

Destructive Distillation Products.—When subjected to dry distillation by heating over a gas flame "without boiling," poppy oil yields a consistent oleaginous substance which partly solidifies on cooling, and has a faintly acid reaction, but does not cede to water any trace of sebacic acid. If, when distillation ceases, a fresh receiver be provided, and the heat increased, the oil begins to "boil," gives off acrolein, and an oily distillate comes over which gives up to water an acid liquid containing sebacic and acetic acids. By stopping the distillation when half the oil has passed over the residue left in the retort forms, when cold, a thick, viscid mass, slightly coloured, and possessing many of the properties of the anhydride of linoleic acid, which easily melts when warmed.

Besides the solid glycerides of stearic and palmitic acid, poppy-seed oil contains oleic, linoleic (in large quantity) and linolenic acids.

Percentage of Free Fatty Acids.—The oil contains varying amounts of free fatty acids. The following percentages were found by various observers: Rechenberg, 2·09; Salkowski, 2·29. Nordlinger found: (*a*) in 26 samples of expressed salad oil 0·70 to 2·86, average 1·92; (*b*) in 5 expressed commercial samples 12·87 to 17·73, average 15·37; (*c*) in 5 extracted samples 2·15 to 9·43, average 4·72.

Poppy-seed oil is not often adulterated, but when sophisticated it is generally with sesame, which can be detected by the lower iodine value and the Badouin reaction.

FATTY ACIDS.

	Benedikt and Ulzer.	De Negri and Fabris.	Hübl.	Thorner.
Melting-point Degrees C. -	—	20·21	20·5	20·5
Solidification Point - -	—	—	16·5	16·5
Saponification Value - -	—	—	—	199
Mean Molecular Weight -	279·1	—	—	—
Iodine Value - - -	—	139	—	116·3
Acetyl Value - - -	13·1	—	—	—

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POPPY-SEED OIL.

Observer.	Density.		Solidification Point Degrees C.	Hehner's Value.	Saponification Value	Iodine Value.	Maumené's Test. Degrees C.	Index of Refraction.
	Degrees C.							
Allen	·15	0·924-0·937	—	—	—	—	—	—
Archbutt	98-99	0·8738	—	—	—	—	86-88	—
Beckurts and Seiler	—	—	—	—	—	—	—	72 at 25°
Clarke	15	0·9262	—	—	—	—	—	C. (Zeiss's
Crossley and Le Sueur	15·5	0·9255-0·9268	—	94·97	189-196 8	133·7-137·1	—	Inst.)
Dietzel and Kreszner	—	—	—	95·38	—	—	—	63·4 at 40°
De Negri and Fabris	15	0·927	—	—	193·6	136·8-137·6	87-88·5	(Zeiss)
Dieterich	—	—	—	—	197·7	137·6-143	—	—
Gerard	—	—	−18	—	—	—	—	—
Hübl	—	—	—	—	—	136	—	—
Jean	—	—	—	—	—	—	—	+23-35
Mansfeld	—	—	—	—	—	—	—	74·5 at 25°
Maumené	—	—	—	—	—	—	74	C. (Zeiss's
Moore	—	—	—	—	192·8	134	—	Inst.)
Shukoff	—	—	—	—	—	—	—	—
Souchere	·15	0·9245	−17-19	—	—	134-135	—	—
Stillwell	18	0·9245	—	—	—	—	—	—
Thorner	—	—	—	—	193-194	134-135	—	—
Ulzer	—	—	—	—	—	138·6	—	—
Valenta	—	—	—	—	194·6	—	—	—

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Walnut Oil.

This oil, known as nut oil, is a *drying* oil, and must not be confounded with other *non-drying* nut oils. It is obtained from the kernel of the common walnut, *Juglans regia.* (Juglans = *Jovis glans* or *Jove's acorn.*)

Artists' Oil.—The kernels are peeled and pressed in the cold, when they yield a rather cloudy oil difficult to clarify, of a pale yellowish green tint, equal to, if not superior, in drying propensities to linseed oil, with an agreeable smell and nutty flavour.

Virgin Oil.—The walnut kernels are not peeled, but the fully matured nuts are stored for two or three months until decomposition sets in, when the kernels are pressed, yielding in the cold 25 per cent. of their weight of virgin oil. The oil expressed in this manner is used as an edible oil, and as a substitute for linseed oil.

Second Quality Oil.—The marc from the first pressing is ground with hot water and again pressed, and the dark inferior oil, with an acrid taste and smell, thus obtained is used as a paint oil.

Walnut oil dries to a more elastic film or coat, with less tendency to crack than linseed oil. For fine, delicate and white colours the better qualities of walnut oil are highly prized by artists on account of their being almost colourless, but their cost is too great for general use.

Genuine walnut oil is difficult to procure. To make sure of its purity it must be extracted from the nuts by those who want to use it. The Smithsonian Institute, Washington, purchased the following samples as genuine walnut oil:—

No. 1. *Walnut Oil, White.*—This article was colourless, of sweetish taste, with a peppermint-like flavour, and soluble in water and 92 per cent. and 50 per cent. alcohol. Further investigation showed it to be diluted glycerine, flavoured with a menthol-like body.

No. 2. *Walnut Oil, Conc.*—The word concentrated immediately cast a halo of suspicion about this oil, and, on submitting it to a fractional distillation, about 80 per cent. came over between 78° and 85° C., which was chiefly ethyl alcohol. Then the thermometer rose rapidly to 205° C., which is the boiling-point of nitrobenzine (oil of mirbane), and the odour confirmed the boiling-point. A small amount of non-volatile matter was left.

When it is remembered that oil of walnuts is chiefly used by artists for paints, because it dries into a varnish which is less liable to crack than linseed oil varnish, the enormity of such adulterations becomes self-evident.

Having been unable to secure an oil of good quality, walnut kernels were secured, ground, and the oil expressed by means of a hydraulic press. In this way 25 per cent. of oil was obtained, while the kernels actually contained 66 per cent. of oil. It was thus deemed of interest to investigate the oil, inasmuch as no such examination seems to have been made.

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The oil generally used is that obtained from *Juglans regia, L.*, a tree indigenous to Persia, and cultivated in Europe and America. The kernels of this nut contain from 30 per cent. to 40 per cent. of "virgin" oil. The fresh cold drawn oil is limpid, nearly colourless or pale greenish yellow, and of agreeable taste and odour, has a specific gravity of 0·925 to 0·9265 at 15° C., saponification number 186-197, iodine value 142 to 151·7, fusing-point of fatty acids 16° to 20° C., dries well, and is said to be brought into U.S.A. from France and Switzerland in 110-gallon tuns.

Hickory-nut oil resembles the above walnut oil very much, and is known as "American nut oil".

Wm. T. Brant says "oil of black walnuts is sometimes expressed, but is of little value". On examining the cold pressed black walnut oil, the following physical and chemical constants were obtained. It is limpid, of a straw-yellow colour, possesses a pleasant, agreeable, walnut-like odour and taste, becomes turbid at 12° C., has a specific gravity of 0·9215 at 15° C., saponification number 190·1-191·5, acid number 8·6-9, ether number 181·5-182·5, Hehner's number 92·77, Reichart-Missel value 15 c.c., iodine value 141·4-142·7, melting-point of fatty acids 0° C.

The drying qualities are excellent, equal, if not superior in this respect, to linseed oil, leaving a tenacious, flexible, transparent film. An artist, on using it, pronounced it a very satisfactory article for fine painting.

By a German process patented by Th. C. Graham, I. H. Kellog and W. K. Kellog, D.R.P. 109,237, the oil is extracted from the nuts by roasting them between 150° and 180° C., 302° and 356° F., and the shelled kernel made into a paste and mixed with water at 55° C. (32 litres of water to 100 kilogrammes of nuts when the oil floats to the surface).

FATTY ACIDS.

Observer.	Melting-Point Degrees C.	Solidification Point.	Mean Molecular Weight.	Iodine Value.	Acetyl Value.
Benedikt and Ulzer - -	—	—	273·5	—	7·6
De Negri and Fabris - -	16-18	—	—	150·05	—
Hübl - - - - -	20	16	—	—	—
Kebler - - - -	16-20	—	—	—	—

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WALNUT OIL.

Observer.	Density.		Solidification Point.	Saponification Value.	Iodine Value.	Maumené Test. Degrees C	Refractive Index.
	Degrees C						
Allen - - - -	15	0·925-0·926	Thickens at – 15° C.	—	—	—	—
Crossley and Le Sueur	15·5	0·9259	Solidifies at – 27·5° C.	192·5	143·1	101	64·8 in Zeiss's Inst.
De Negri and Fabris -	15	0·9265	—	193·81-197·92	144·5-145·1	96	—
Dieterich - - - -	—	—	—	188·7	147·9-151·7	—	—
Hazura - - - -	—	—	—	—	145·7	—	—
Hübl - - - -	—	—	—	—	143	—	—
Jean - - - -	—	—	—	—	—	—	+ 35- + 36
Kebler - - - -	15	0·925-0·9265	—	186-197	142-151·7	—	Jean's
Maben - - - -	—	—	—	194·4	—	—	Inst.
Maumené - - - -	—	—	—	—	—	101	—
Peters - - - -	—	—	—	—	143·3	—	—
Petkow - - -	15	0·9255-0·9260	—	—	147·9-148·4	—	67·68 in Zeiss's Inst.
	12	0·928	—	—	—	—	
Saussure - -	25	0·919	—	—	—	—	—
	94	0·871	—	—	—	—	—
Souchere - - -	15	0·926	—	—	—	—	—
Valenta - - -	—	—	—	196	—	—	—

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